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ADVANCED THERMOPLASTIC RESINS - PHASE II

A. M. Brown, S. G. Hill, and A. Falcone

BOEING DEFENSE & SPACE GROUP
Aerospace & Electronics Division
Seattle, Washington 98124

Contract NAS1-17432
September 1991

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Langley Research Center
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FOREWORD

This report describes the work accomplished under Contract NAS1-17432, Phase II, "Advanced Thermoplastic Resins." The contract was sponsored by the National Aeronautics and Space Administration, Langley Research Center, Hampton, Virginia 23665-5225.

Mr. Paul M. Hergenrother was the NASA Technical Monitor. The Materials and Processes Technology organization of the Boeing Defense Group - Aerospace and Electronics Division was responsible for the work performed. Ms. Arlene M. Brown was Program Manager. Mr. Sylvester G. Hill was Technical Leader and Mr. Anthony Falcone was principal investigator. The following personnel provided critical support to the program.

Oscar N. Davis	Mechanical Testing
Paul B. Y. Mori	Adhesive and Titanium Preparation
Frank D. Horan	Composite Fabrication

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1.0 Summary and Introduction

1.1 Summary

NASA-Langley developed polyimide resins were evaluated as high temperature structural adhesives for metal to metal bonding and as composite matrix resins. Adhesive tapes were prepared on glass scrim fabric from solutions of polyamic acids of a semicrystalline polyimide, designated LARC-CPI and a thermoplastic polyimide, designated LARC-TPI. Good bond strengths were obtained at ambient temperature and acceptable strengths were obtained at elevated temperature. Composite properties of LARC-TPI were lower than desired and are compared to some other thermoplastic composites.

LARC-CPI (Langley Research Center - Crystalline Polyimide) was supplied by NASA as polyamic acid solutions (25 weight percent solids) in N,N-dimethylacetamide (DMAc). Adhesive tapes were prepared by priming Style 112 E-glass scrim having an A-1100 finish with polyamic acid solution diluted with DMAc, and drying at 200°C or 250°C. Coats of the polyamic acid solution were applied undiluted and dried individually at the same temperature to produce a tape 0.30 to 0.36 mm (12 to 14 mils) thick. Drying at these temperatures converts most of the polyamic acid to polyimide by cyclodehydration, and removes reaction byproducts and residual solvent.

Titanium (6Al-4V) lap shear specimens were prepared by grit blasting and treating the bonding surfaces with Pasa-Jell 107 (Semco, Inc.) which creates a conversion coating on the titanium and titanium oxide surfaces. Five-finger panels were assembled in fixtures and autoclave bonded. Lap shear specimen assembly and testing were conducted per Boeing Support Specification 7202.

The LARC-CPI resin was very processable, yielding low volatile content adhesive tapes with adequate flow, and titanium lap shear bond strengths of up to 45.2 MPa (6550 psi) at ambient and 14.0 MPa (2030 psi) at 232°C using a bonding pressure of 1.38 MPa (200 psi). Elevated temperature bond strengths were improved by aging at 316°C. Higher bond strengths at elevated temperature were reported by NASA resulting from differences in adhesive tape preparation, test specimen geometry, and test machine operation.

LARC-TPI (Langley Research Center - Thermoplastic Polyimide) resin powders were evaluated for titanium honeycomb core bonding. The imidized powders from Mitsui Toatsu Chemicals and Rogers Corporation were produced by chemical imidization of the polyamic acid to produce a fine powder. Slurries of the powders in Rogers' polyamic acid solution in diglyme were used to prepare adhesive tapes which were dried at 200°C. The titanium face sheets and honeycomb core were treated with Pasa-Jell 107, primed with dilute polyamic acid solution, and dried at 200°C. The primed titanium honeycomb core (6.35 mm cell size) was dip coated with the powder slurry and dried at 200°C as well. Sandwich assemblies of tape between core and face sheets were bonded at 0.31 MPa (45 psi) and 343°C for 30 minutes.

LARC-TPI resin powder slurries worked well as titanium honeycomb core bonding adhesives. The keys to obtaining satisfactory bond strengths were to evenly coat the core ribbon with sufficient resin for filleting yet thinly enough to avoid trapping volatiles, and to use a bonding temperature that was high enough to obtain adequate resin flow. Flatwise tensile strengths of up to 4.62 MPa (670 psi) and 2.90 MPa (420 psi) were obtained at test temperatures of ambient and 232°C at a bonding pressure of 0.31 MPa (45 psi). There was a significant variation in the lot to lot processing parameters of the LARC-TPI powders. Some powders required higher bonding temperatures and pressures than others to obtain suitable bond strengths.

Composite laminates were fabricated in both the press and autoclave using prepreg produced from a 1:1 slurry of LARC-TPI powder in a polyimidesulfone ($PISO_2$) polyamic acid solution in diglyme with 5 weight percent of a bisamideacid (BAA) added to enhance resin flow and composite processability. The carbon fiber reinforcement was unsized Hercules AS4 which was treated just prior to prepregging with BAA to facilitate handling of the fibers.

Although the mechanical test results obtained from polyimidesulfone/LARC-TPI composites were comparable to results obtained earlier and were comparable to some high temperature thermoplastic composites they were not as high as desired. Average zero degree tensile and compressive strengths of 1,290 MPa (187 ksi) and 883 MPa (128 ksi) respectively were obtained at ambient temperature. LARC-TPI powders with more consistent melt flow properties are needed to repeatably obtain low void content laminates. A moderate degree of polymer adhesion to unsized AS-4 carbon fibers was observed in scanning electron micrographs of fractured test coupons.

1.2 Introduction

High temperature structural resins are required for use on advanced aerospace vehicles as adhesives and composite matrices. Although most adhesive and composite applications in commercial aircraft involve lower operating temperatures with the exception of limited areas around the engines, there is increasing interest in high speed civil transport (HSCT) aircraft which will require materials that can withstand tens of thousands of hours of operation at temperatures in the range of 150 to 204°C (300 to 400°F), as well as exposure to water, hydraulic fluid, fuel, and deicing fluid in some areas. Work on commercial supersonic transport aircraft (SST) first took place in the 1960s.

The higher HSCT operating temperatures preclude the use of aluminum for the structure which means that some combination of titanium and composites will be employed in the aircraft structure. The desired combination of long range and large passenger capacity for the HSCT aircraft may cause designers to select composites for large sections of the airplane's structure. However, for whatever design is developed, high temperature service resins will be required for bonding and for composite matrices.

Uncertainty about the HSCT aircraft speed and therefore operating temperatures and durations at those temperatures, coupled with the long lead times necessary for materials selection and qualification, causes engineers to be conservative in selecting candidate materials. Materials with higher temperature capabilities than might be absolutely necessary may need to be selected, due to these design uncertainties, so that the requisite testing and qualification can be completed in time for full scale engineering development.

Military aircraft also require higher service temperatures due to aerodynamic heating at their higher speeds. An example is the interest in thermoplastic and bismaleimide composites for 177°C (350°F) service in the Air Force Advanced Tactical Fighter program.

In missile applications higher temperature polymeric materials are required for tactical, strategic, and large launch systems. Applications include fairings, body sections, nose caps, and fins. Lower cost composite materials and manufacturing methods have become increasingly important as the cost of these and other military aerospace vehicles have increased. Adhesives and composites are required for space vehicles that will cycle thousands of times between -160 to 120°C (-250 and 250°F), and exhibit low outgassing and resistance to radiation. Polymer adhesives and composites for individual aerospace applications require a unique set of properties including high strength and stiffness, toughness, high glass transition temperature, solvent resistance, and sufficiently low viscosities (high flow) for processing.

Many polyimide polymers have excellent high temperature properties well suited for aerospace structural applications, however, they are often challenging to synthesize and process as adhesives and composites matrices. Polyimide chemistry and properties have been extensively investigated and reviewed (Ref. 1). Polyimides are often synthesized by a condensation reaction in solution polymerization by the reaction of an aromatic dianhydride with an aromatic diamine to form a polyamic acid, which is then converted to the polyimide by thermal or chemical cyclodehydration. Many factors such as addition of excess reactant, the speed of addition of a reactant, and the solvent used affect the equilibrium reaction in which the polyamic acid is formed, and hence determine the properties of the final polyimide product. Side reactions also take place which can influence the properties of the resulting polyimide.

Polyimide molecular weight can be controlled by the addition of excess dianhydride or diamine to the polyamic acid solution. The molecular weight and distribution can be controlled by the addition of end-cappers, such as aniline or naphthalic anhydride, which prevent further chain extension. End-cappers can also reduce the evolution of volatiles (and void formation) during subsequent processing by preventing chain extension. The amount and type of end-capper can also influence the degree of crystallinity (Ref. 2)

High molecular weight polyimide resins often exhibit inadequate flow under the usual pressures employed for adhesive bonding and composites processing. Resin flow is important for wetting adherends in bonding, and for wetting fibers and fusing plies together in composite laminate fabrication. Controlling polyimide molecular weight and molecular weight distribution to enhance resin flow during processing, and improving batch-to-batch consistency is an ongoing effort.

The properties and processability of a polyimide are, of course, also a function of the polymer molecular structure. For example meta oriented diamines appear to result in improved processability (flow) over para oriented diamines (Ref. 3). A carbonyl linkage in the anhydride (BTDA) results in crystalline polyimide while the increased flexibility resulting from an ether linkage in the anhydride (ODPA versus BTDA) appears to result in an amorphous polyimide (Ref. 4).

The solvents used in polyimide synthesis and processing also significantly influence polyimide adhesive and composite properties. Polar, aprotic solvents are used for polyimide synthesis because they form strongly hydrogen bonded complexes with the free carboxyl groups of the polyamic acid, shifting the equilibrium towards the polyamic acid product. Removal of the high boiling point organic solvents can be difficult, and can cause void formation in adhesive bondlines or composite laminates. Ether solvents appear to enhance the adhesive properties of polyimides, and bis(2-methoxy-ethyl) ether (diglyme) is a better wetting agent for titanium than amide solvents (Ref. 5).

The LARC-CPI (Langley Research Center - Crystalline Polyimide) and LARC-TPI (Langley Research Center - Thermoplastic Polyimide) condensation polyimide resins are produced from polyamic acid solutions by thermal or chemical imidization. The polyamic acid prepolymer solutions are easily handled and can be used to coat glass fabric to produce adhesive tape or to coat graphite fibers to produce composite prepregs, and are then dried to largely complete the imidization and remove solvent and reaction byproducts. The degree of conversion of the polyamic acid to polyimide is a function of time and temperature. The tape is used for bonding under applied heat and pressure, and the prepreg can be laid up and consolidated under heat and pressure in an autoclave, for example. LARC-CPI has a glass transition temperature (Tg) of about 222°C and a crystalline melt temperature of approximately 350°C. LARC-TPI exhibits a Tg of about 250°C. The chemistry and properties of LARC-CPI (Ref. 2, 4, and 6-10) and LARC-TPI (Ref. 11-13) have been published.

The objective of this program was to assess the potential of NASA-Langley developed advanced thermoplastic resins for structural use in aerospace vehicles under high service temperatures. LARC-CPI was of interest because of its high toughness, tensile strength and modulus, glass transition temperature (Tg), good solvent resistance, and processability due to its crystalline melting behavior. Crystallinity imparts good solvent resistance, and high strength and stiffness at elevated temperature. LARC-TPI was of interest because of its excellent adhesive properties under long term ageing, and the development of some semicrystalline enhanced flow grades. Originally two adhesives and two composite materials were to be evaluated. However, one polyimide composite material designated BTDA/BAMC/DAT polyimide was dropped from the program because an adequate quantity of prepreg could not be obtained from the polymer solution in meta-cresol.

2.0 Adhesives

Structural adhesives for metal to metal bonding are typically applied as supported film adhesives. An adhesive tape is prepared by applying a resin solution and oven drying each coat. The metal adherends are surface treated, primed with a dilute solution of the adhesive resin, dried, assembled in a fixture with the tape sandwiched between the metal adherends, and bonded together under heat and pressure. LARC-CPI and LARC-TPI were evaluated as adhesives for 6Al-4V titanium bonding in these tasks.

2.1 LARC-CPI Resin for Titanium Bonding

2.1.1 Resin Composition

The LARC-CPI resin solution in N,N-dimethylacetamide (DMAc) was supplied at 25 percent solids content by NASA-Langley. The resin solution had an inherent viscosity of 0.6 dL/g (0.5 percent concentration in DMAc at 25°C). The resin solution was stored under a nitrogen blanket at 4°C (40°F). This version of LARC-CPI had a stoichiometry upset by 6 mole percent in favor of 3,3',4,4'-benzophenonetetracarboxylic dianhydride (BTDA). Four mole percent of phthalic anhydride had been added as an end-capper to improve the melt stability (Ref. 2).

2.1.2 Adhesive Tape Preparation

Adhesive tapes were prepared from the resin solution by applying and drying successive coats on Style 112 E-glass scrim having an A-1100 finish (γ–aminopropylsilane). The E-glass scrim was clamped between two aluminum picture frames having a 30.5 cm square opening. The scrim was stored at -18°C.

The E-glass scrim was first dried for 30 minutes at 107°C (225°F) and then both sides of the scrim were brush coated with a primer solution prepared from a 3:1 dilution with DMAc of the LARC-CPI resin solution. The primer was allowed to dry in air under a chemical fume hood for at least one hour, and then dried at 250°C for 30 minutes in a forced air oven according to the drying cycle in Figure 1. The dwell at 150°C was intended to allow much of the DMAc (boiling point of 166°C) to diffuse from the tape at a slow

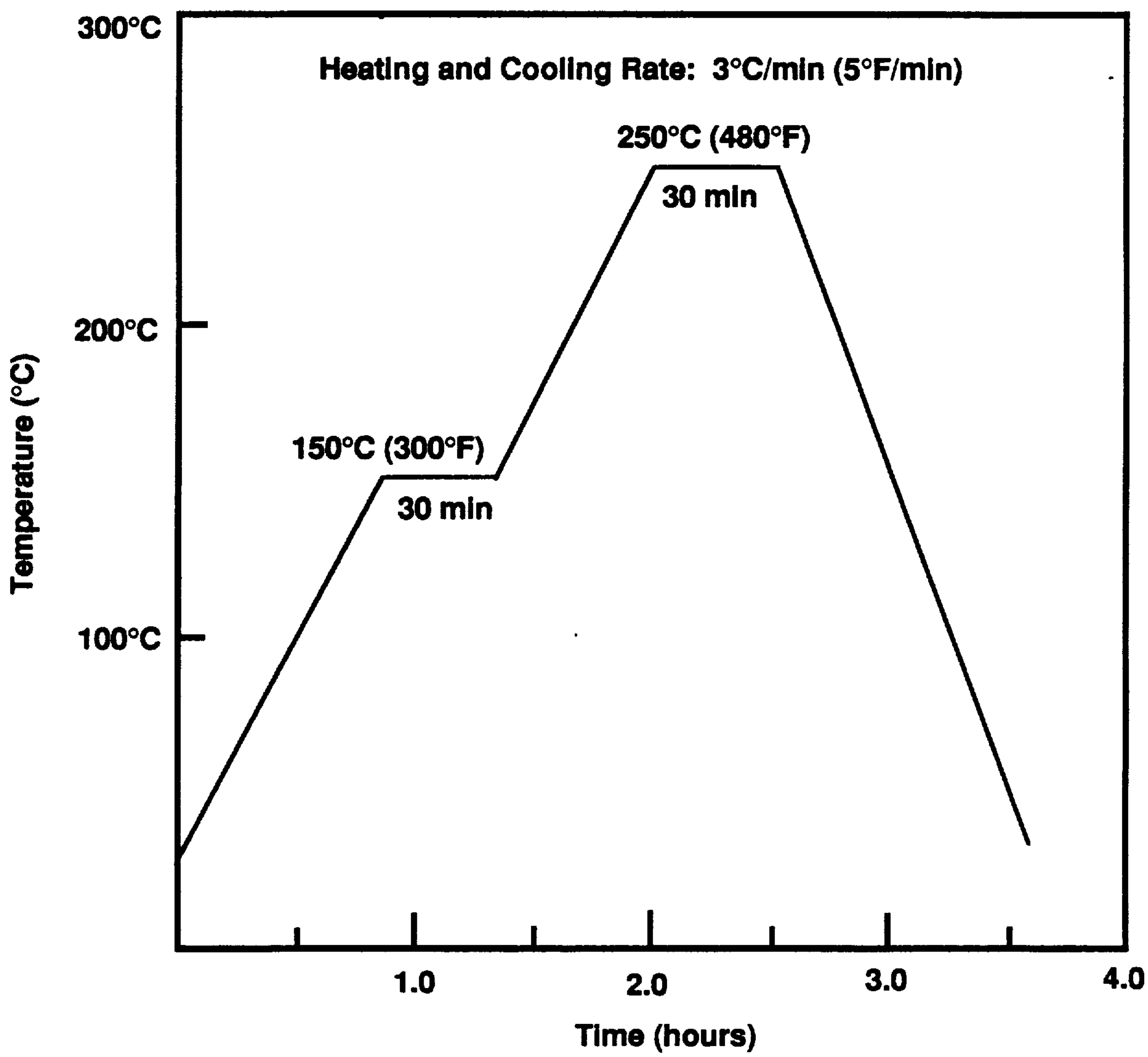

Figure 1. Drying Cycle for LARC-CPI Adhesive Tape

enough rate to avoid the formation of blisters or fish eyes. A second coat of primer was then applied to the tape and dried.

Coats of the undiluted resin solution were then put on one side of the scrim at a time using a plastic sweep (Figure 2) and dried using the same drying schedule as for the primer (Figure 1). Each coat was allowed to air-dry in the hood for at least one hour or overnight prior to oven drying. Six to eight coats of LARC-CPI resin solution were required to produce an adhesive tape that was 0.30 to 0.36 mm (12 to 14 mils) thick. The adhesive tape was stored in a sealed polybag to avoid moisture absorption.

Although a drying temperature of 200°C was also used to prepare some tapes, the higher drying temperature of 250°C resulted in higher lap shear bond strengths. After the tape was prepared additional drying at 250°C was sometimes necessary to reduce the volatiles content to the desired level between 0.3 and 1.0 percent. Drying largely converts the polyamic acid to polyimide, and removes the reaction byproducts (such as water) and solvent from the tape.

The LARC-CPI resin was readily processable and easy to work with. Uniform coats were easy to apply and the resin did not have a tendency to blister or crack. A tape was lost only once, when the resin turned milky white which may have resulted from moisture absorption. Adhesive tapes were stored at ambient temperature in sealed polybags until bonding, and were sealed in polybags if more than a day passed between coats.

Adhesive tapes were analyzed for volatiles content and degree of flow. Volatiles content determination was a straightforward weight loss measurement: the tape was dried at 343°C (650°F) for thirty minutes and the weight loss was measured. The target volatiles content was between 0.3 to 1.0 weight percent.

If a tape had too high a volatiles content the resin would char during bonding and the resulting bonds would have low strength. With too low of a volatiles content (less than 0.1 percent) the resin did not flow enough to wet out the surface. Low volatile contents between 0.3 to 1.0 percent resulted in the best bond strengths. Higher volatile contents resulted in darkening of the resin and poor bond strength with largely adhesive failures.

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Figure 2. Application of LaRC-CPI Resin Solution to E-glass Scrim With Plastic Sweep

Adhesive tape flow was characterized by using a 3.6 cm (1.4 in) diameter disc cut from the tape and sandwiched between two layers of Kapton film coated with Frekote 700 to allow the specimens to be separated from the film. The films and tape disc were placed between steel or aluminum caul plates and heated and compressed in a platen press for 30 minutes at the bonding temperature 400°C (750°F) under the bonding pressure 1.38 MPa (200 psi). The weight of resin that had flowed out beyond the disc circumference was then measured and reported as a weight percentage or percent of resin flow.

2.1.3 Titanium Surface Preparation

Pasa-Jell 107 conversion coating solution was selected to prepare the titanium surfaces for bonding. Chromic acid anodizing was undesirable because of the toxic chromium compounds used in this process and because the high bonding temperatures were known to degrade anodized surfaces. Also, there was a sizeable database for bonded titanium test specimens prepared with Pasa-Jell 107. Titanium adherends 1.27 mm (0.050 in) thick of 6Al-4V titanium were used for lap shear specimens.

The NASA-Langley developed procedure for titanium surface preparation using Pasa-Jell 107 was followed and is outlined below:

1. The adherends were washed with methanol.

2. The adherends were abraded with a grit blast of 100 mesh alumina at a pressure of 0.55 MPa (80 psi).

3. The wash with methanol was repeated.

4. The adherends were dipped in Pasa-Jell 107 for one minute, then removed and allowed to etch for 10 minutes (Figure 3).

5. The one minute dip in Pasa-Jell 107 followed by the 10 minute etch was repeated.

6. The adherends were rinsed in hot water for 5 minutes and then suspended in a tap water ultrasonic bath for 5 minutes.

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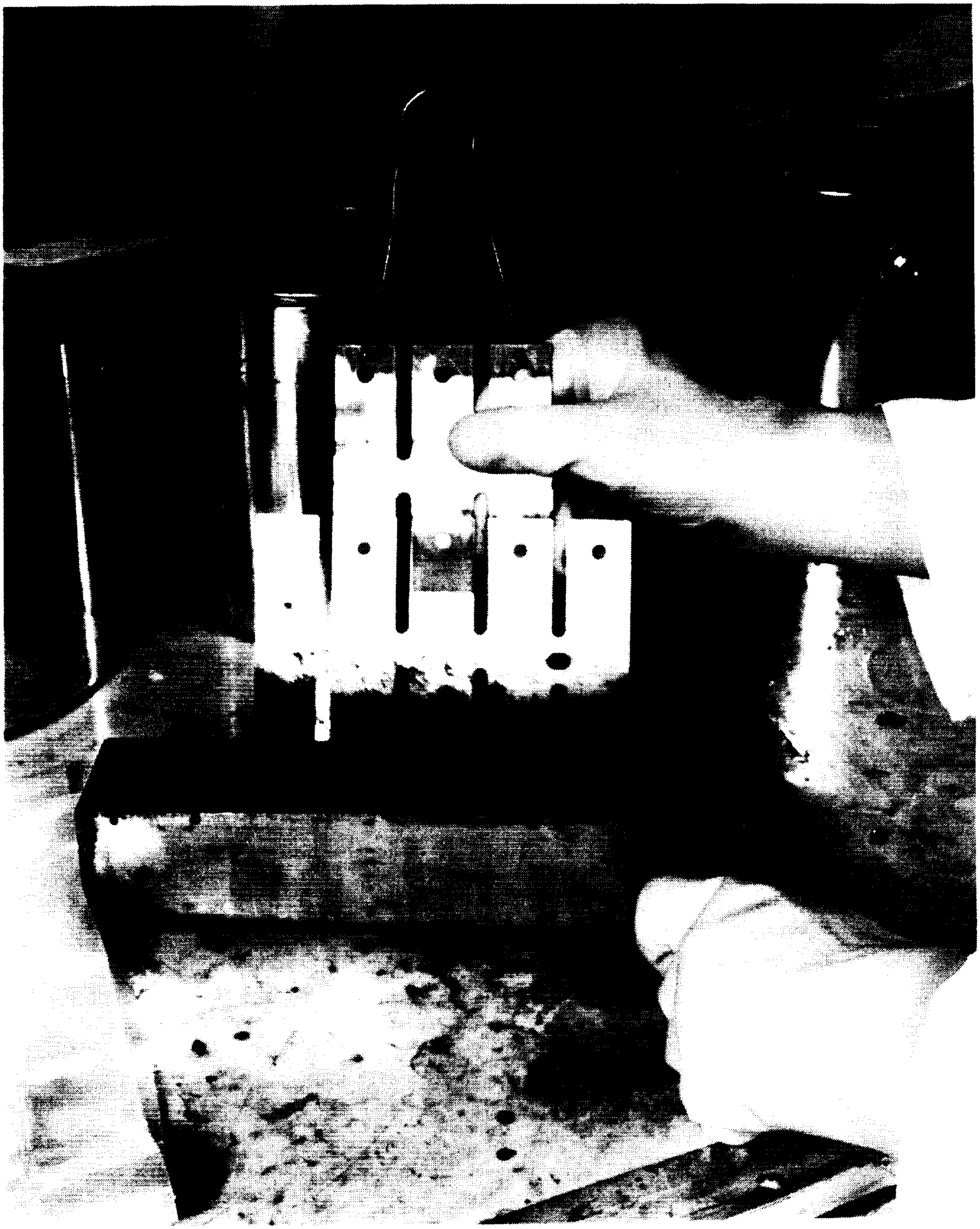

Figure 3. Dipping Titanium Lap Shear Finger Panel in Pasa-Jell 107

7. After air drying for 5 minutes, the adherends were rinsed in an ultrasonic water bath of deionized water for 5 minutes.

8. After drying in air for 10 minutes at room temperature, the adherends were held in an oven at 70°C (158°F) for 10 minutes.

9. The adhesive primer (3:1 dilution of resin solution with solvent) was applied to the adherends within 4 hours.

The primer on the adherends was dried at the same temperature as the tape (250°C).

2.1.4 Adhesive Bonding

Bonding of test specimens was usually performed in the high temperature autoclave since better pressure distribution is obtained in autoclave bonding, and it was more efficient for producing larger numbers of specimens than was a platen press. The same bonding fixture was used for press bonding with a bar to apply clamping pressure.

Titanium finger panels were bonded per Boeing Support Specification 7202 (Ref. 14). The bonding fixture (Figure 4) was vacuum bagged with Kapton film (Figure 5).

2.1.5 Test Results - Including a Comparison with NASA Data

The initial lap shear test results that were obtained with LARC-CPI adhesives are described in this section along with comments on the bonding process. The bonding process includes the adhesive tape volatiles content, degree of resin flow, the bonding temperature, pressure, time, the resulting bondline thickness, and any postcuring or heat treating steps performed on the bonded assemblies.

Testing was performed in a 22 thousand inch-pound Instron Universal Test Machine (Figure 6). Figure 7 shows a titanium lap shear specimen mounted in the grips within the environmental chamber which was used to heat specimens for elevated temperature

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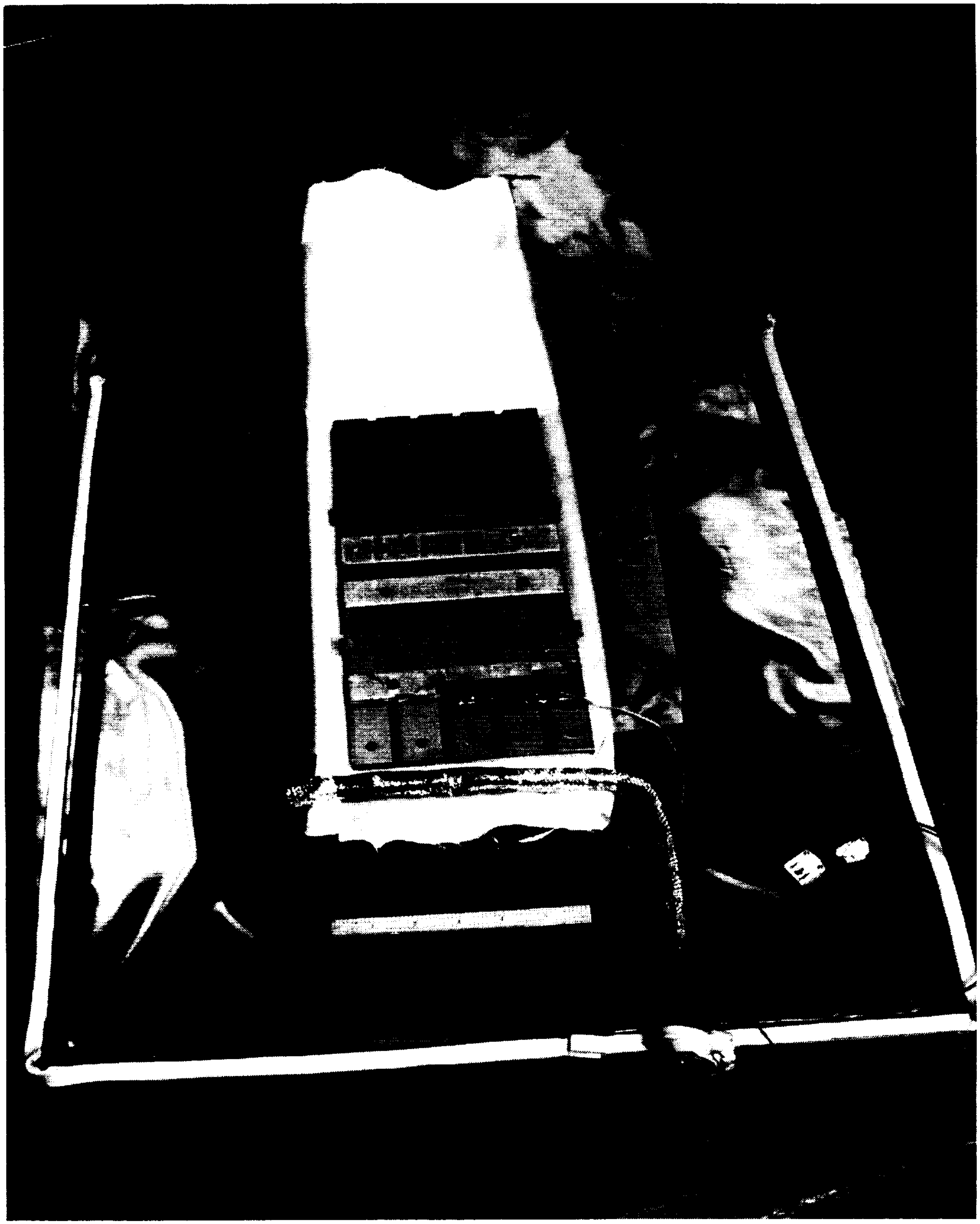

Figure 4. Titanium Lap Shear Finger Panels and Adhesive Tape in Bonding Fixture Before Vacuum Bag Was Sealed

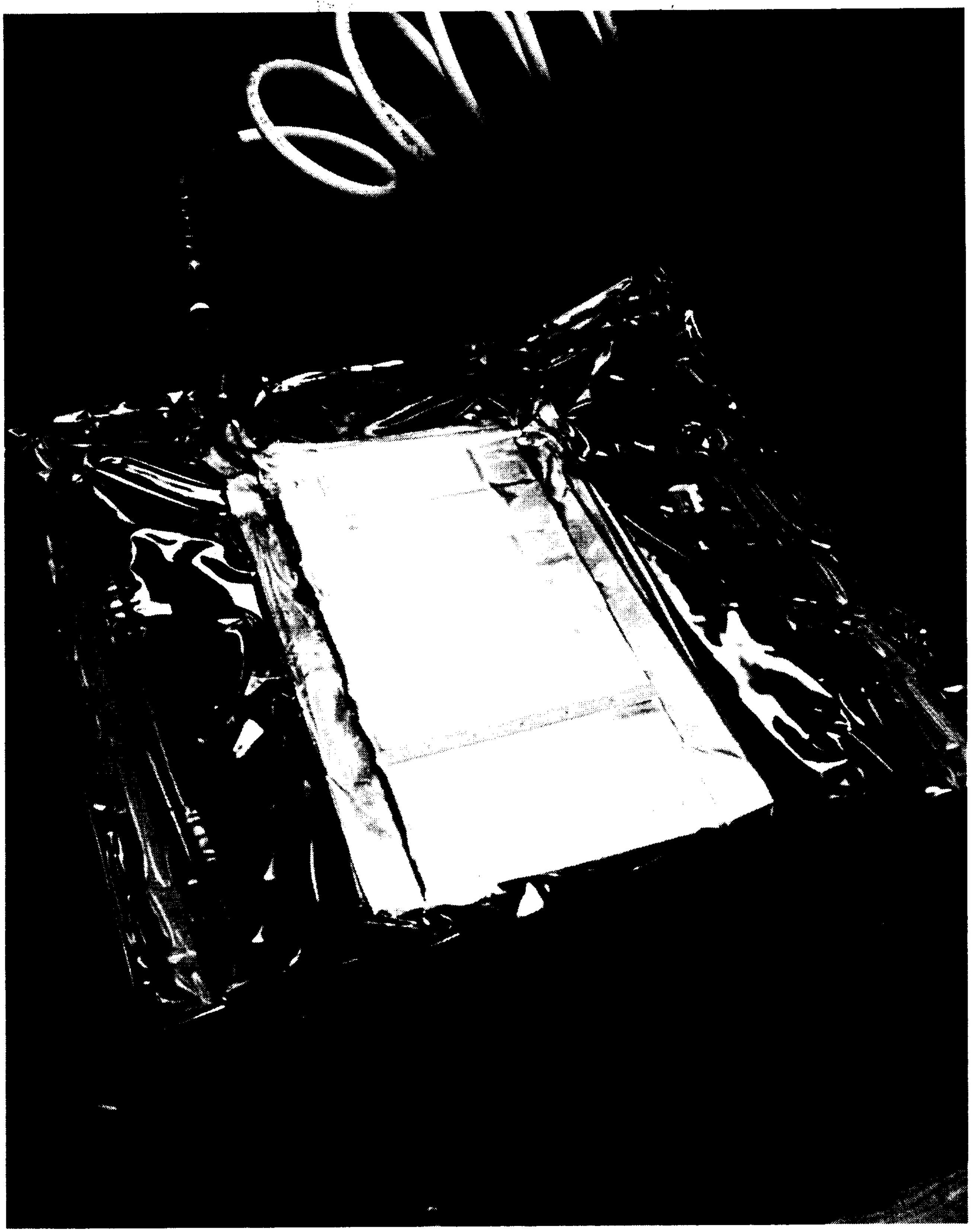

Figure 5. Titanium Lap Shear Bonding Fixture Sealed in Vacuum Bag Ready for Autoclave Bonding

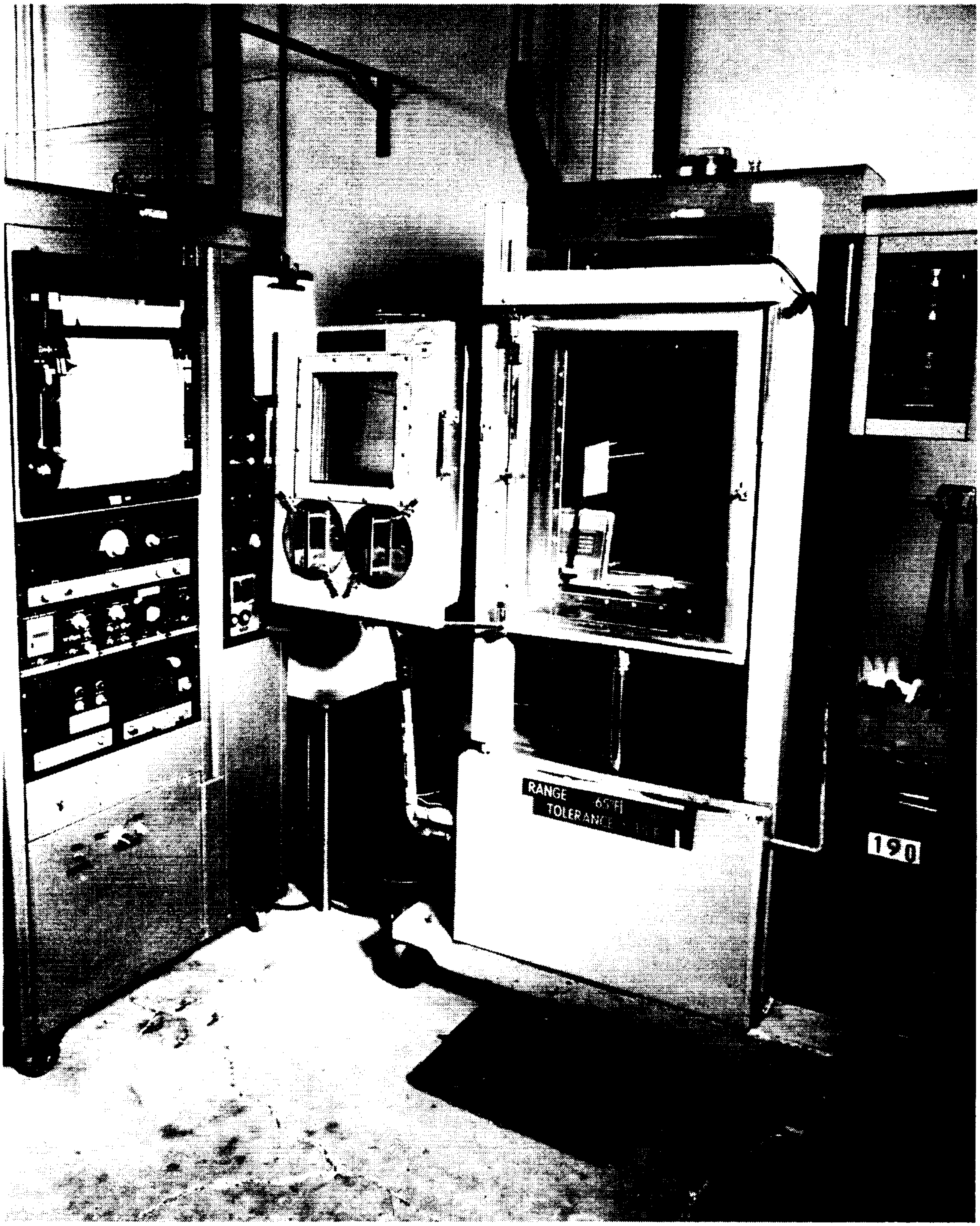

Figure 6. Instron Universal Testing Machine With Environmental Chamber

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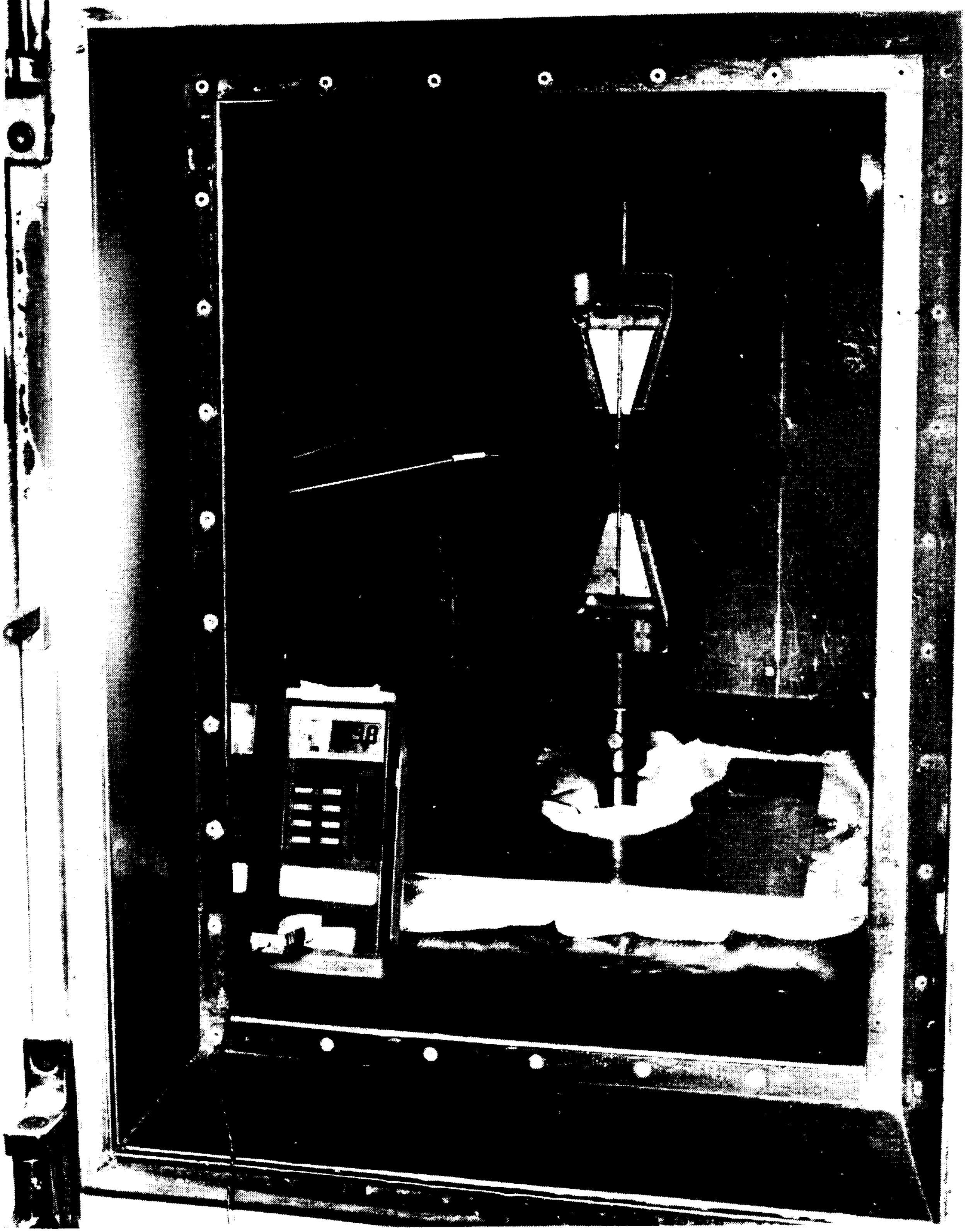


Figure 7. Titanium Lap Shear Specimen Mounted in Grips of Instron Within the Environmental Chamber

testing. A thermocouple was clipped on the specimen with a spring loaded clamp.

The lap shear strengths at elevated temperatures obtained at Boeing were lower than elevated temperature strengths obtained by NASA, although initial ambient temperature strengths were comparable. Adhesive tapes and test specimens were exchanged with NASA in an effort to determine why lap shear strengths measured by Boeing were low. Surface analysis of the bonding and fracture surfaces and analysis of the lap shear testing configuration are discussed.

The following tables contain summaries or average test values. Test values measured for individual specimens are listed in the tables of Appendix A which reference tables in this report.

2.1.5.a) Initial Results

Adhesive tapes were prepared and 6Al-4V titanium lap shear specimens were bonded at selected values of the bonding process variables described previously. Tapes were prepared from LARC-CPI at drying temperatures of 200 and 250°C and dried to various volatile contents (Table I and Figure 8). As the volatiles content of the tape increased, the degree of flow generally increased. Good adhesive flow is necessary for obtaining high bond strengths. However, the volatiles must be effectively removed from the bond during processing to prevent void formation and resin charing which lower bond strengths.

Titanium lap shear specimens were bonded at 400°C (750°F) which is above the crystalline melting temperature of LARC-CPI, and bonding pressures were limited to pressures that could be obtained in standard autoclaves. A bonding pressure of 1.38 MPa (200 psi) resulted in satisfactory bond strengths and could be attained in standard autoclaves. Bond strengths increase or decrease as the bonding pressure is increased or decreased. Increased bonding pressure causes more adhesive flow, better infiltration and wetting of the surface conversion coating or oxide layer, and therefore enhanced bonding.

Titanium lap shear strengths (Table II) were highest for tapes having low volatile contents (less than one percent after drying for 30 minutes at 343°C). Average lap shear strengths of 32.2 MPa (4670

Table I. Volatiles Content and Degree of Flow - LARC-CPI Adhesive Tapes - Pressure Applied at 400°C (750°F)

Specimen	Volatiles Content (wt%)	Applied Pressure MPa (psi)	Time min	Degree of Flow (wt%)
1	2.9	6.90 (1000)	30	49.5
2	2.9	1.38 (200)	30	44.4
3	2.9	1.38 (200)	15	48.0
4	2.3	1.38 (200)	15	57.8
5	0.95	1.38 (200)	15	22.4
6	1.4	1.38 (200)	15	41.0
7	0.34	1.38 (200)	15	34.7

Notes:
1. Specimens 1-3 dried at 200°C.
2. Specimen 4 dried an additional 11 hours at 200°C
3. Specimen 5 dried an additional 110 hours at 200°C
4. Specimen 6 dried at 250°C
5. Specimen 7 dried at 250°C, then dried an additional 72 hours at 250°C.

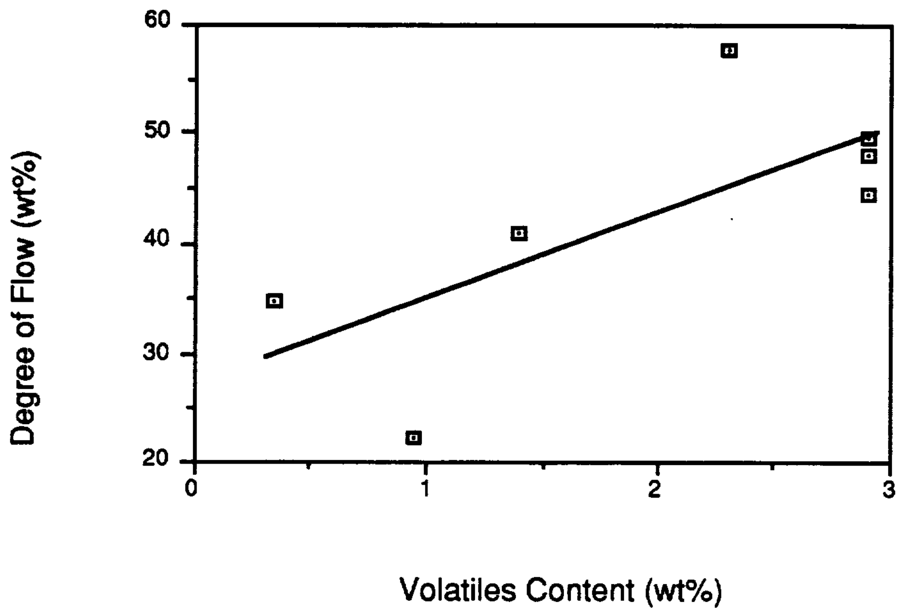


Figure 8. Volatiles Content Versus Degree of Flow for LARC-CPI Adhesive Tapes, With Pressure Applied at 400°C (750°F) for 30 Minutes

psi) were obtained at 1.4 percent volatiles versus 25.7 MPa (3730 psi) at 2.3 percent volatiles. With further drying to lower volatile contents (0.34%), higher bond strengths of up to 45.2 MPa (6550 psi) at ambient temperature and up to 14.0 MPa (2030 psi) at 232°C were obtained as summarized in Tables III and IV, and Figures 9, 10, and 11.

Table II. LARC-CPI Room Temperature Adhesive Bond Lap Shear Strengths as a Function of Tape Volatiles Content

Volatiles Content (wt%)	Average Shear Strength MPa (psi)	Failure Surface
2.9%	22.7 (3290)	80% Adhesive
2.3%	25.7 (3730)	70% Adhesive
1.4%	32.2 (4670)	90% Cohesive

Bonding temperature of 400°C (750°F) at 6.90 MPa (1000 psi). Bonding pressure was high due to an equipment malfunction.

The three figures (9, 10, and 11) illustrate the effects of volatiles content, heat treating, and test temperature on titanium/LARC-CPI bonds. Figure 9 shows that as the adhesive tape volatiles content was reduced the bond strength increased. In Figure 10 elevated temperature bond strengths were improved by post bonding heat treatment although at a sacrifice in ambient temperature strength. Note also that elevated temperature bond strength began to decline with heat treating at 316°C for more than 100 hours. With 500 hours of heat treatment at 316°C the lap shear strength decreased from 16.2 to 5.41 MPa at ambient and from 14.0 to 4.32 MPa at 232°C. Specimens from this group were not tested at a time between 100 and 500 hours of heat treatment. Figure 11 shows the decrease in lap shear strength as a function of test temperature; at 232°C the LARC-CPI resin is slightly above its glass transition temperature of 222°C, however, the crystalline polymer regions should still carry appreciable loads (Ref. 2).

Table III . Summary of Initial LaRC-CPI 6A1-4V Titanium Lap Shear Strengths

Specimen Numbers	Wt% Volatiles	Wt% Flow	Bonding Pressure MPa (psi)	Bondline Thickness (mils)	Test Temperature °C (°F)	Average Strength MPa (psi)	COV	Failure Surface**
250-1A-1	1.37	41.0	6.90 (1000)	2.5 - 3.5	Room Temp.	32.2 (4670)	0.04	90-100% C
250-1B-1	0.34	34.7	1.38 (200)	2.8 - 4.5	Room Temp.	45.2 (6550)	0.09	100% C
250-1B-2	0.34	34.7	1.38 (200)*	3.0 - 5.0	232 (450)	13.2 (1910)	0.18	50-90% C
250-2A-2	0.12	11.1	0.69 (100)*	3.5 - 5.5	Room Temp.	20.7 (3000)	0.10	95% C
250-2A-1	0.12	11.1	0.69 (100)*	4.0 - 5.0	177 (350)	15.2 (2210)	0.11	100% C
250-2A-3	0.12	11.1	0.69 (100)*	3.5 - 4.5	200 (392)	15.0 (2180)	0.11	100% C
250-2A-4	0.12	11.1	0.69 (100)*	3.5 - 5.0	232 (450)	11.7 (1700)	0.10	95-100% C
250-2A-5	0.12	11.1	1.38 (200)*	3.0 - 4.3	Room Temp.	34.4 (4990)	0.04	95% C
250-2A-6	0.12	11.1	1.38 (200)	3.8 - 4.5	Room Temp.	38.3 (5560)	0.05	100% C
250-2A-7	0.12	11.1	1.38 (200)	3.0 - 4.0	Room Temp.	36.9 (5350)	0.13	100% C
250-2A-8	0.12	11.1	1.38 (200)	3.5 - 5.0	232 (450)	4.30 (620)	0.12	100% A
200-2A-1	2.9	44.4	6.90 (1000)	3.0 - 5.0	Room Temp.	22.7 (3290)	0.26	100% A
200-2B-1	2.3	57.8	6.90 (1000)	6.0 - 7.0	Room Temp.	25.7 (3730)	0.13	100% C
200-2C-1	0.95	22.4	1.38 (200)	3.0 - 4.0	Room Temp.	44.7 (6480)	0.03	50-90% C
200-3A-1	0.93	22.6	1.38 (200)*	7.5 - 8.7	232 (450)	3.46 (500)	0.42	50-100%A

*Postcured at 300°C (570°F) and 0.69 MPa (100 psi) for 4.0 hours. **C = Cohesive, A = Adhesive.

Table IV. LARC-CPI Titanium Lap Shear Strengths

Drying Temp. (°C)	Vol. (wt%)	Flow (wt%)	Bond Press MPa	Test Temp. (°C)	Average Strength MPa (psi)	Failure Surface
250	0.71	26.6	1.38 2/	ambient	39.3(5700)	100% Coh
250	0.34	34.7	1.38 3/	ambient	16.2(2350)	95% Adh
250	0.34	34.7	1.38 3/	232°C	14.0(2030)	40-70% Coh
250	0.12	11.1	1.38 4/	ambient	5.41(785)	95% Adh
250	0.12	11.1	1.38 4/	232°C	4.32(626)	95% Adh

Notes:

1. LARC-CPI resin solution batch number 51388.
2/ Postcured for 4 hours at 300°C and 0.69 MPa.
3/ Postcured for 4 hours at 300°C and 0.69 MPa, followed by heat treatment for 100 hours at 316°C and atmospheric pressure.
4/ Postcured for 4 hours at 300°C and 0.69 MPa, followed by heat treatment for 500 hours at 316°C and atmospheric pressure.

Air flow rate in the heat treatment oven was 4.13 m^3/min (146 ft^3/min).

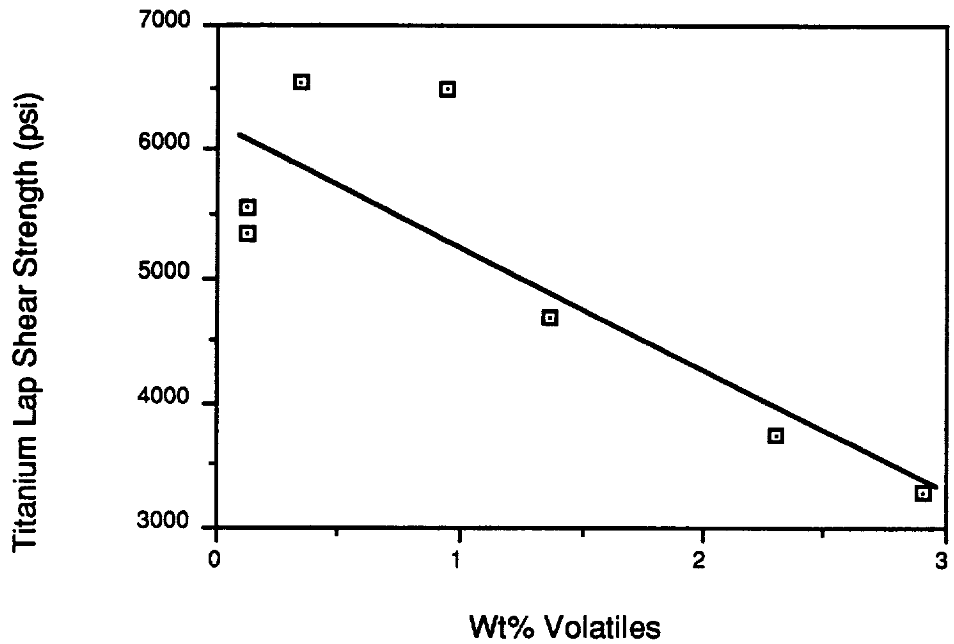


Figure 9. LARC-CPI Adhesive Tape Volatiles Content Versus Bond Strength Bonding was performed at 1.38 MPa (200 psi), specimens were not postcured, and specimens were tested at ambient temperature.

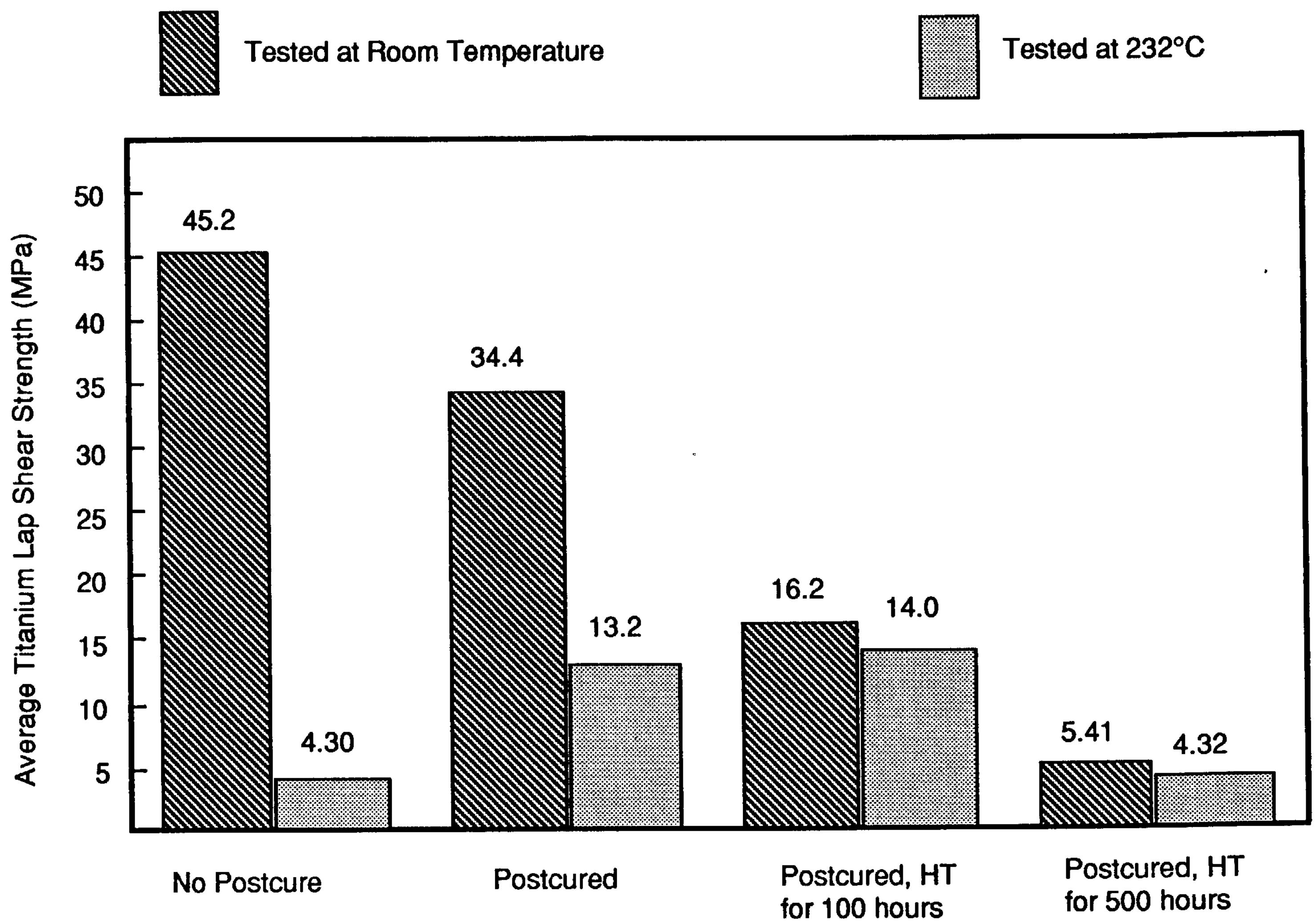


Figure 10. Comparison of Average Titanium Lap Shear Strengths With and Without Postcuring and Heat Treatment (HT) at Room Temperature and 232°C

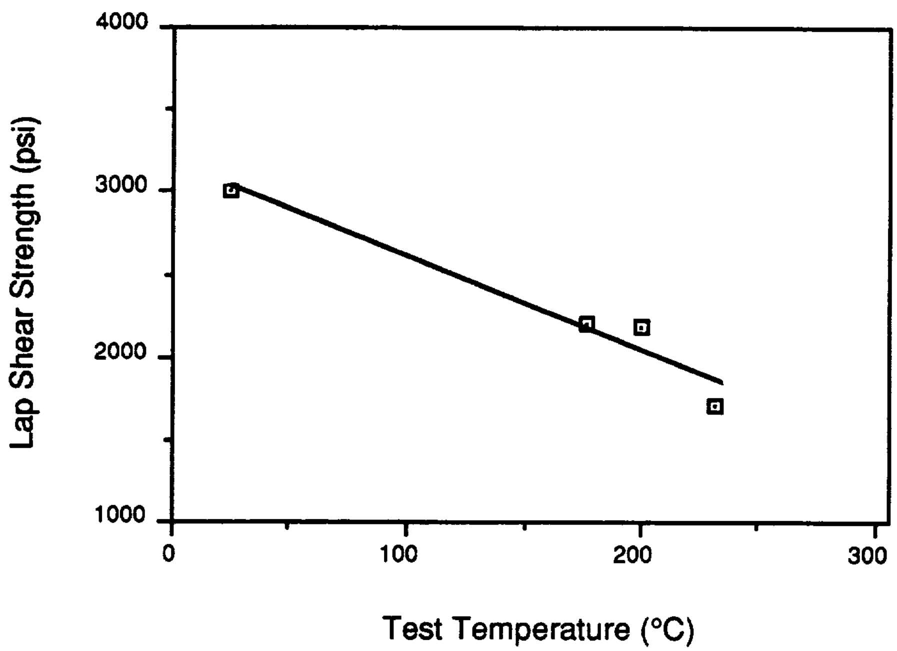


Figure 11. Titanium Lap Shear Strength as a Function of Temperature. Tape volatiles: 0.12 wt%; Degree of Flow: 11.1; Bonding Pressure: 0.69 MPa (100 psi). Postcured at 300°C and 0.69 MPa for 4.0 hours.

Ambient temperature lap shear strength was reduced slightly by postcuring at 300°C but was greatly reduced by the heat treatment at 316°C. The term postcuring as used here refers to holding the specimens at elevated temperature under pressure. Heat treatment was performed at atmospheric pressure in a circulating air oven (air flow rate in the heat treatment oven was 4.13 m^3/min (146 ft^3/min)). The failure surfaces of the ambient temperature tested specimens changed from cohesive to adhesive upon heat treatment at 316°C. Although the lap shear strengths at ambient temperature were greatly reduced, the lap shear strengths at 232°C remained about the same or were slightly improved by heat treatment at 316°C. It has been suggested that the heat treatment may be allowing recrystallization to occur accompanied by slight shrinkage. As a result, the resin may adhere more tightly to the titanium conversion coating/oxide layer of the Pasa-Jell 107 treated specimens (Ref. 2).

Comparing the ambient and 232°C test specimens treated for 100 hours at 316°C (Table IV), a slight drop in lap shear strength was observed (16.2 to 14.0 MPa) along with changes in the failure surfaces. At ambient temperature the failure was mainly adhesive while at 232°C it was largely cohesive. Silver-gray titanium metal was visible on the ambient temperature test specimen fracture surfaces while the olive-green primed surface was visible on the elevated temperature test specimens. As the test temperature was increased the failure appeared to occur between the primer and adhesive rather than between the primer and the titanium or titanium conversion coating. In specimens that were postcured but not heat treated, failure appeared to occur within the resin and primer for specimens tested at both ambient and elevated temperature.

With further treatment at 316°C to 500 hours the average lap shear strength decreased to about one third the value measured after 100 hours. The failure surfaces were adhesive with failure in the primer around the edges of the bond area and silver-gray titanium metal in the center of the bond area. This mixed-mode failure surface occurred in the specimens tested at ambient temperature and at 232°C after 500 hours at 316°C.

Although the heat treatment at 316°C appeared to increase lap shear strength at elevated temperature, the strength at ambient temperature was greatly reduced from 16.2 to 5.41 MPa and failure occurred between the primer and titanium (or perhaps between the conversion coating and the titanium). Similar results were obtained for titanium lap shear specimens prepared with another adhesive tape (0.51% volatiles, 26.1% flow, Table A8) on a Boeing process development project for high temperature adhesives.

Photographs of several high strength titanium lap shear specimens tested at ambient and 232°C appear in Figures 12 and 13. Note that the scrim fabric was distorted due to the resin flow during bonding and that these bonds had high shear strengths due primarily to high adhesive flow.

2.1.5.b) Adhesive Tapes and Test Specimens Exchanged with NASA

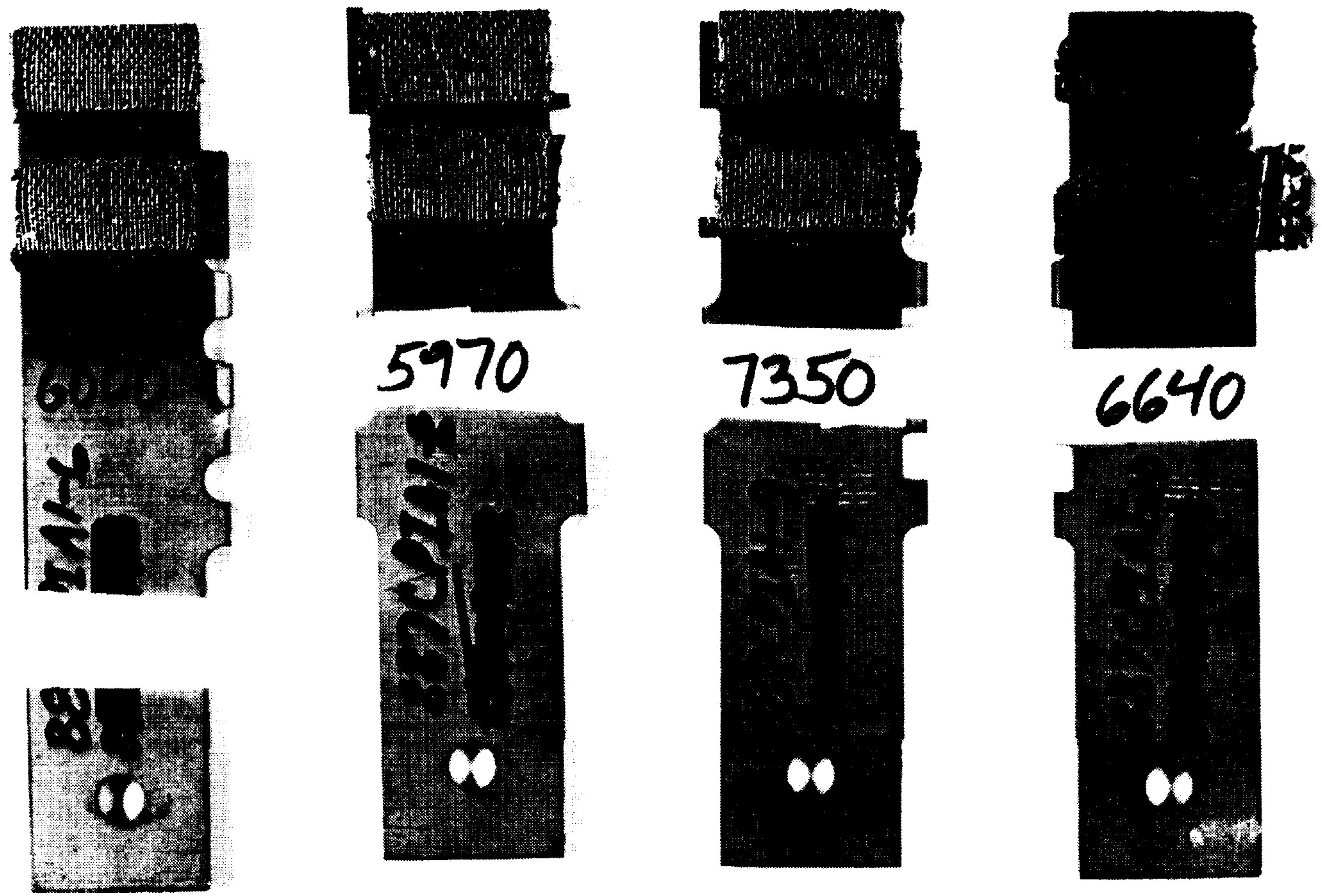
A titanium lap shear strength of 17 MPa (2500 psi) at 232°C (450°F) or better was desired using a maximum bonding pressure of 1.38 MPa (200 psi). The highest average shear strength obtained at Boeing was 14.0 MPa (2030 psi) at 232°C. NASA-Langley reported titanium lap shear strengths of up to 24.1 MPa (3500 psi) at 232°C under the same bonding conditions. Although excellent lap shear strengths were obtained initially at ambient temperature by Boeing [up to 45.2 MPa (6550 psi) average lap shear strength], elevated temperature lap shear strengths were not as high as NASA had obtained.

The discrepancy in elevated temperature test results could result from differences in tape preparation, titanium surface preparation, bonding procedure, or mechanical testing procedures. To determine why Boeing elevated temperature bond strengths were not as high as NASA's, the following exchanges were performed.

i) Sending adhesive tape that had been prepared at Boeing to NASA; NASA used the tape to bond lap shear specimens which were then tested at NASA,

ii) Sending surface treated and primed lap shear specimens to NASA for bonding with NASA-prepared adhesive tape. The specimens were then tested at Boeing,

iii) NASA sent lap shear specimens prepared from their tape to Boeing for testing.



887CPIA1 - 6 TO 10
VOL = 0.34%   FLOW = 34.7%
POST CURE: NONE   HEAT TR = NONE
TEST: AMBIENT   LSS = 6550 PSI   100% COHESIVE

Figure 12. Titanium Lap Shear Specimens Tested at Ambient Temperature; Average Lap Shear Strength of 45.2 MPa (6,550 psi)

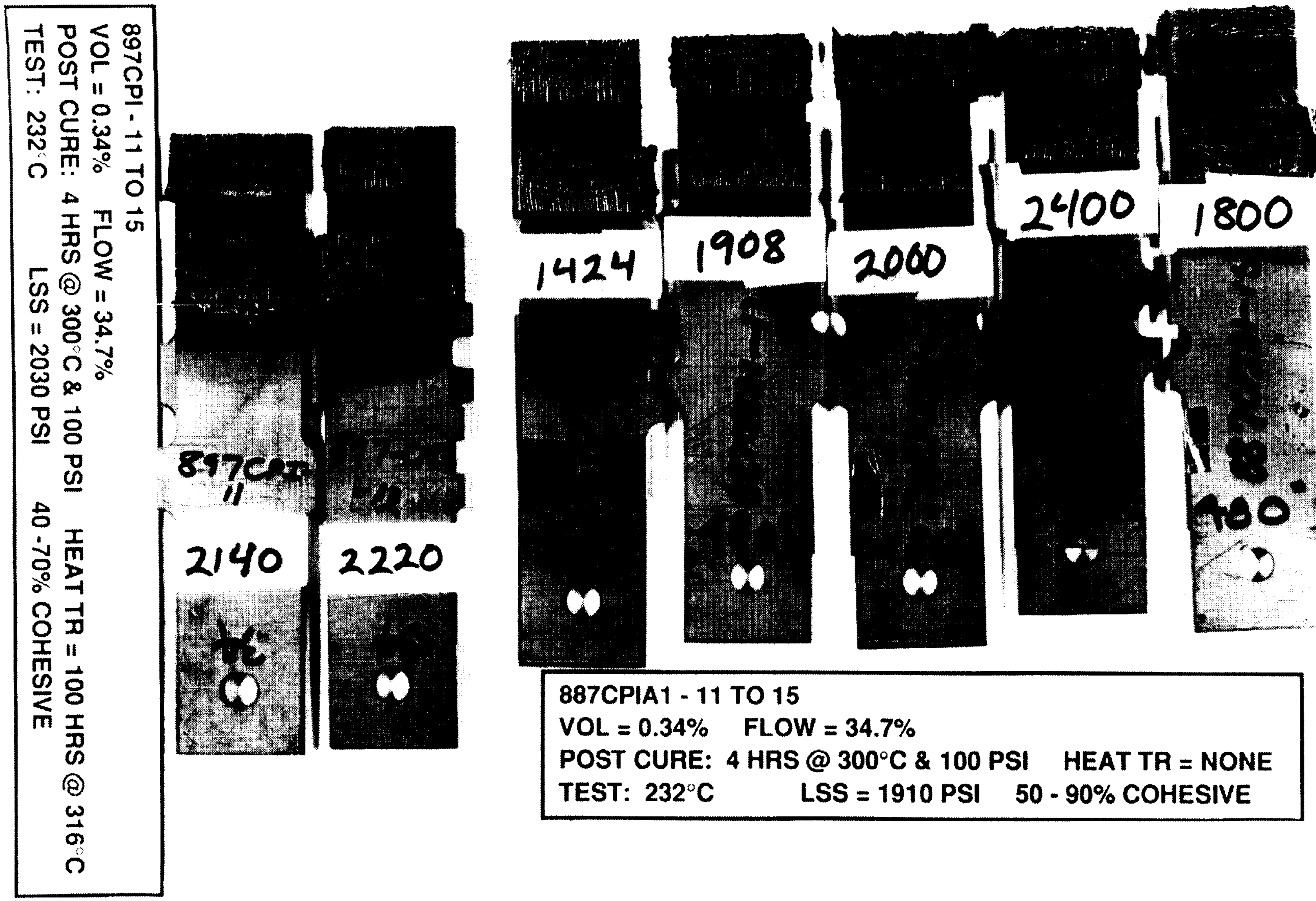


Figure 13. Titanium Lap Shear Specimens Tested at 232°C; Average Lap Shear Strength of 14.0 MPa (2,030 psi) and 13.2 MPa (1,910 psi)

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i) Adhesive Tape Sent to NASA

To resolve the discrepancy in test results at elevated temperature a sample of LARC-CPI adhesive tape prepared at Boeing was used to fabricate bonds at Boeing and also at NASA. The tape was prepared from LARC-CPI resin solution batch number SH 39-92-17 (inherent viscosity of 0.57 dL/g) and had a volatiles content of 0.51 wt% and a degree of flow of 26.1 wt%. The coats of LARC-CPI resin were dried at 250°C. A volatile content determination performed by NASA-Langley by heating for 18 hrs at 300°C showed 0.88 wt% volatiles. The Boeing volatiles content determinations were performed by heating for 30 minutes at 343°C (650°F).

The titanium was treated with Pasa-Jell 107 and primed with dilute LARC-CPI resin solution. The specimens were bonded by NASA at 375°C (707°F) for 15 minutes under a pressure of 2.76 MPa (400 psi) although a pressure of 1.38 MPa (200 psi) was intended. The test results from the titanium lap shear specimens bonded and tested at NASA-Langley are shown in Table V.

Two sets (10 lap shear specimens) of titanium lap shear specimens were also bonded at Boeing using the same adhesive tape and bonding temperature. The specimens were treated with Pasa-Jell 107 and primed. The test results are shown in Table VI. The bonding temperature was 375°C for 15 minutes at 1.38 MPa (200 psi). All test results for the individual specimens prepared at both NASA and Boeing are listed in Appendix A. Photographs of the specimens appear in Figures 14 and 15.

Comparing the test results in Tables V and VI and Figure 16, significantly lower lap shear strengths at ambient temperature and at 232°C were obtained at Boeing using the same adhesive tape and the same bonding conditions with the exception of the higher pressure used by NASA. The bonding temperature used by both NASA and Boeing to prepare these specimens was 375°C (707°F) which was lower than the 400°C (750°F) temperature used previously by Boeing.

Table V. Average Lap Shear Strengths of Titanium Lap Shear Specimens Bonded and Tested at NASA-Langley

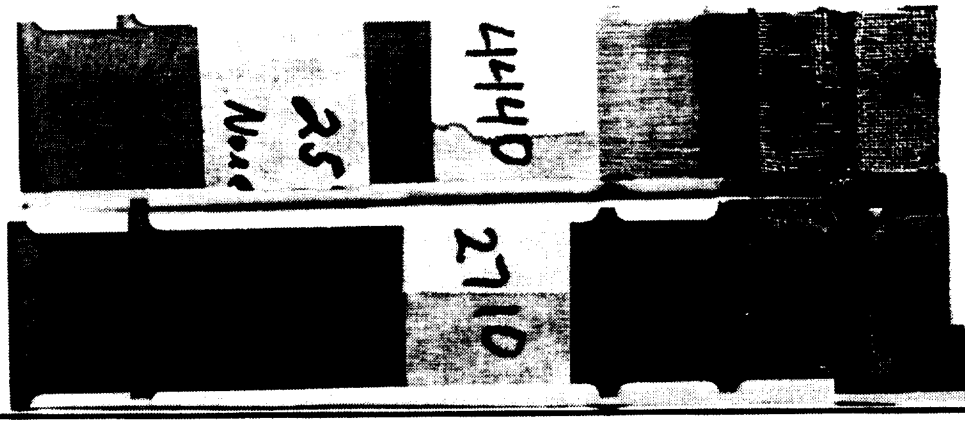
Heat Treatment	Test Temp	Lap Shear Strength* MPa (psi)	Fracture Surface
None	25°C (77°F)	24.7 (3575)	90% Coh/Adh
None	200°C (392°F)	21.9 (3180)	60% Cohesive
None	232°C (450°F)	8.97 (1300)	100% Adhesive
18 hrs @ 316°C	232°C (450°F)	16.1 (2330)	60% Cohesive
100 hrs @ 316°C	200°C (392°F)	22.2 (3230)	90% Cohesive
100 hrs @ 316°C	232°C (450°F)	17.9 (2600)	85% Cohesive

*Average of two specimens.
Boeing adhesive tape, dried at 250°C, 0.51% volatiles, 26.1% flow.
Bonding pressure of 2.76 MPa at 375°C.

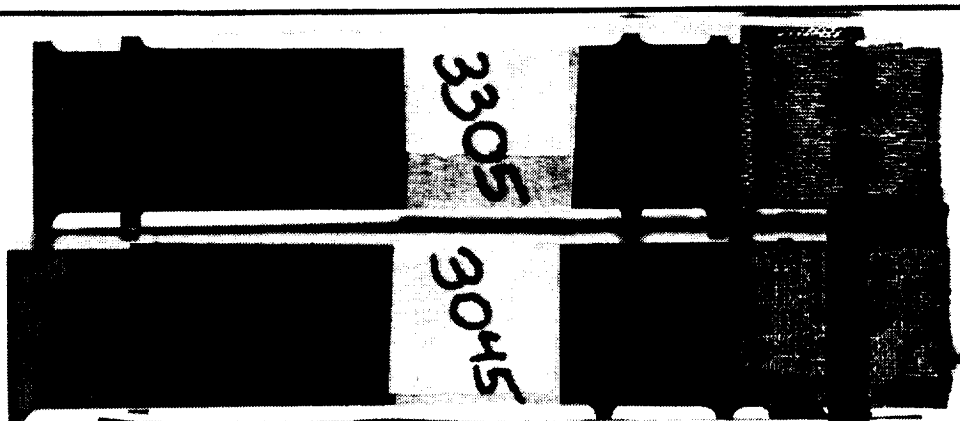
Table VI. Average Lap Shear Strengths of Titanium Lap Shear Specimens Bonded and Tested at Boeing*

Heat Treatment	Test Temp	Lap Shear Strength* MPa (psi)	Fracture Surface
None	25°C (77°F)	6.05 (877)	100% Adhesive
None	232°C (450°F)	6.97 (1010)	100% Adhesive
100 hrs @ 316°C	232°C (450°F)	12.3 (1780)	90% Cohesive

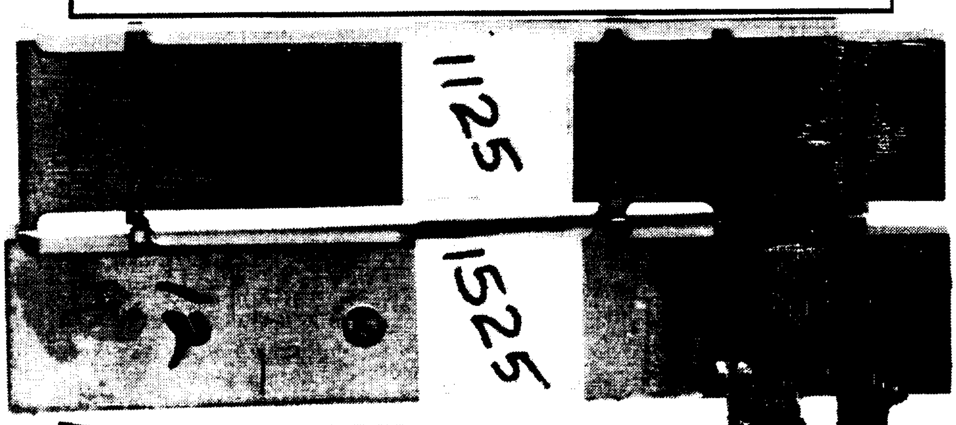
*Average of three specimens.
Boeing adhesive tape, dried at 250°C, 0.51% volatiles, 26.1% flow.
Bonding pressure of 1.38 MPa at 375°C.



NASA: 1 & 6 VOL = 0.51% FLOW = 26.1%
POST CURE: NONE HEAT TR = NONE
TEST: AMBIENT LSS = 4440 & 2710 PSI 90% COHESIVE/90% ADHESIVE

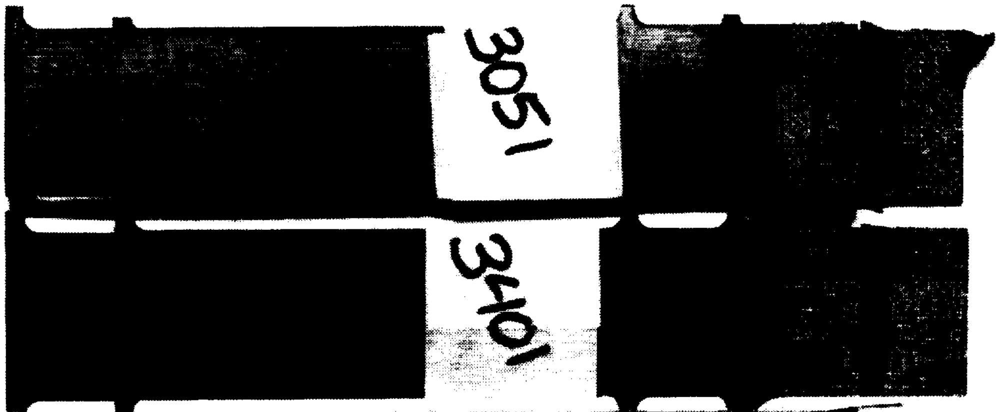


NASA: 2 & 9 VOL = 0.51% FLOW = 26.1%
POST CURE: NONE HEAT TR = NONE
TEST: 200°C LSS = 3175 PSI 60% COHESIVE

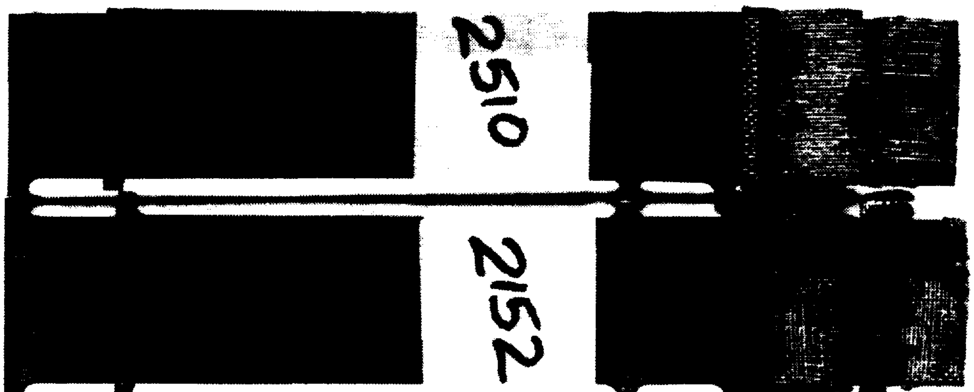


NASA: 3 & 12 VOL = 0.51% FLOW = 26.1%
POST CURE: NONE HEAT TR = NONE
TEST: 232°C LSS = 1300 PSI 100% ADHESIVE

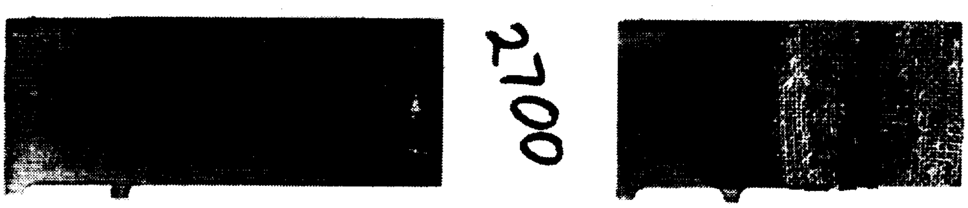
Figure 14. Titanium Lap Shear Specimens Bonded at NASA, With Average Lap Shear Strengths Noted



NASA: 8 & 11 VOL = 0.51% FLOW = 26.1%
POST CURE: NONE HEAT TR = 100 HRS @ 316°C
TEST: 200°C LSS = 3226 PSI 90% COHESIVE



NASA: 4 & 10 VOL = 0.51% FLOW = 26.1%
POST CURE: NONE HEAT TR = 18 HRS @ 316°C
TEST: 232°C LSS = 2331 PSI 60% COHESIVE



NASA: 5 & 7 VOL = 0.51% FLOW = 26.1%
POST CURE: NONE HEAT TR = 100 HRS @ 316°C
TEST: 232°C LSS = 2600 PSI 100%/70% COHESIVE

Figure 15. Titanium Lap Shear Specimens Bonded at NASA, With Average Lap Shear Strengths Noted

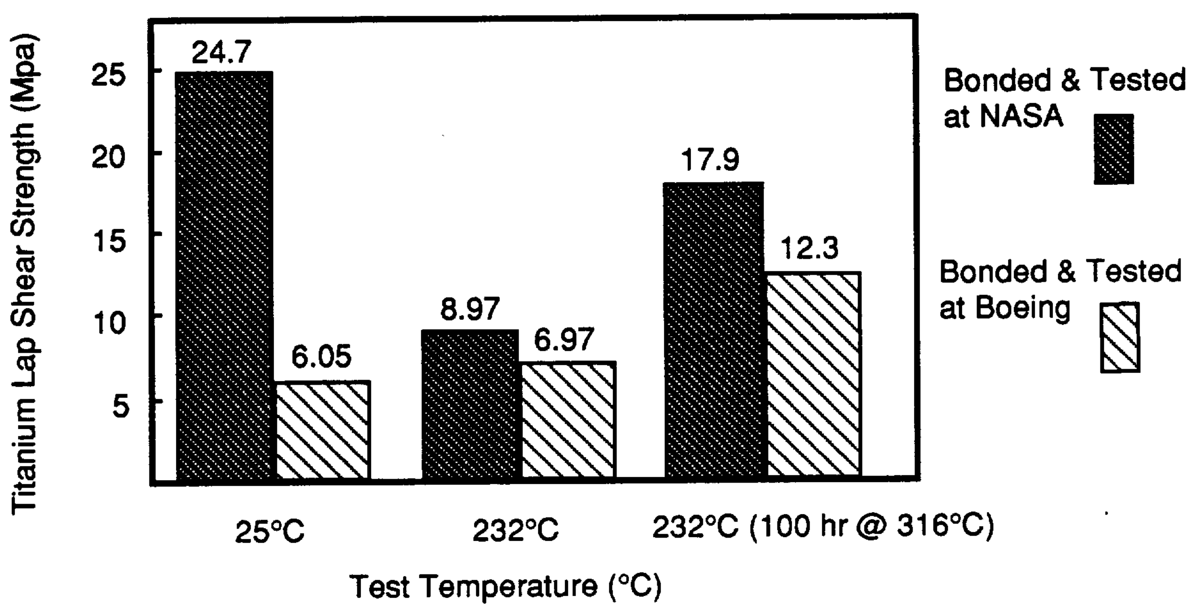


Figure 16. Comparison of Titanium Lap Shear Strengths of Specimens Bonded from Boeing Adhesive Tape Sent to NASA (Tables V and VI). Specimens prepared, bonded, and tested at both NASA and Boeing.

ii) Lap Shear Specimens Sent to NASA

Titanium lap shear specimens that had been treated with Pasa-Jell 107 and primed at Boeing were bonded with LARC-CPI adhesive tape at NASA, and were subsequently tested at Boeing. The test results were compared to the results obtained by NASA using the same adhesive tape with their surface treated titanium.

The average lap shear strengths obtained for the Boeing treated and primed titanium which was bonded at NASA are listed in Table VII. Average lap shear strengths of specimens treated, primed and bonded by NASA using the same bonding conditions are shown in Table VIII. The individual lap shear strength values of the specimens are listed in Appendix A.

The specimens tested at Boeing gave strengths significantly lower than specimens tested at NASA. The bondlines of the first group of specimens that Boeing tested were thick (0.0090 to 0.0105 inches) and in one specimen the corners were not bonded. In the second set of specimens the resin also did not appear to have flowed enough to thoroughly wet out the primer. The lower bonding temperature of 375°C versus 400°C used previously appears to produce bonded specimens with less resin flow. NASA also experienced some difficulty using the bonding fixture that Boeing provided to them which may also have contributed to poorer bonds (thicker bondlines).

After heat treatment of the remaining lap shear specimens, the Boeing lap shear strengths more closely matched the results obtained by NASA at 25 and 200°C and agreed closely with the results of testing at 232°C (Figure 17).

iii) NASA Specimens Received for Testing

Six titanium lap shear specimens bonded with LARC-CPI were received from NASA-Langley. The specimens were bonded with adhesive tape prepared from 30 weight percent polyamic acid solution in diglyme (batch no. SH-69-82-20, inherent viscosity of 0.53 dL/g) dried at 200°C and having a volatiles content of 2.8 weight percent (adhesive tape no. 69-67-1, inherent viscosity of 0.47 dL/g initially and after one week at room temperature under nitrogen the inherent viscosity was 0.39 dL/g). Bonding was

Table VII. Average Lap Shear Strengths of Titanium Lap Shear Specimens Bonded at NASA and Tested at Boeing

Heat Treatment	Test Temp.	Lap Shear Strength MPa (psi)	Fracture Surface
16 hrs @ 308°C	25°C (77°F)	15.6 (2260)	60% Cohesive
16 hrs @ 308°C	200°C (392°F)	12.9 (1870)	60% Adhesive
16 hrs @ 308°C + 100 hrs @ 316°C	25°C (77°F)	19.8 (2870)	80% Adhesive
16 hrs @ 308°C + 100 hrs @ 316°C	200°C (392°F)	22.2 (3220)	60% Cohesive
16 hrs @ 308°C + 100 hrs @ 316°C	232°C (450°F)	11.2 (1630)	80% Adhesive

Average of 5 specimens. Bonded at NASA- Langley using 1.38 MPa (200 psi) and 375°C (707°F), then heat treated at 308°C for 16 hours. Tested at Boeing. Adhesive tape produced at NASA was used to bond all specimens. LARC-CPI resin solution batch number SH 69-67-1. Tape drying temperature: 200°C; volatiles content: 2.8 percent.

Table VIII. Average Lap Shear Strengths of Titanium Lap Shear Specimens Bonded and Tested at NASA.

Heat Treatment	Test Temp	Lap Shear Strength MPa (psi)	Fracture Surface
16 hrs @ 308°C	25°C (77°F)	34.0 (4930)	100% Cohesive
16 hrs @ 308°C	200°C (392°F)	28.1 (4080)	90% Cohesive
100 hrs @ 316°C	25°C (77°F)	26.4 (3830)	80% Adhesive
100 hrs @ 316°C	200°C (392°F)	29.4 (4270)	100% Cohesive
100 hrs @ 316°C	232°C (450°F)	13.3 (1930)	50% Adhesive

Average of 4 specimens. Bonded at NASA- Langley using 1.38 MPa (200 psi) and 375°C (707°F), then heat treated at 308°C for 16 hours. Tested at NASA. Adhesive tape produced at NASA was used to bond all specimens. LARC-CPI resin solution batch number SH 69-67-1. Tape drying temperature: 200°C; volatiles content: 2.8 percent.

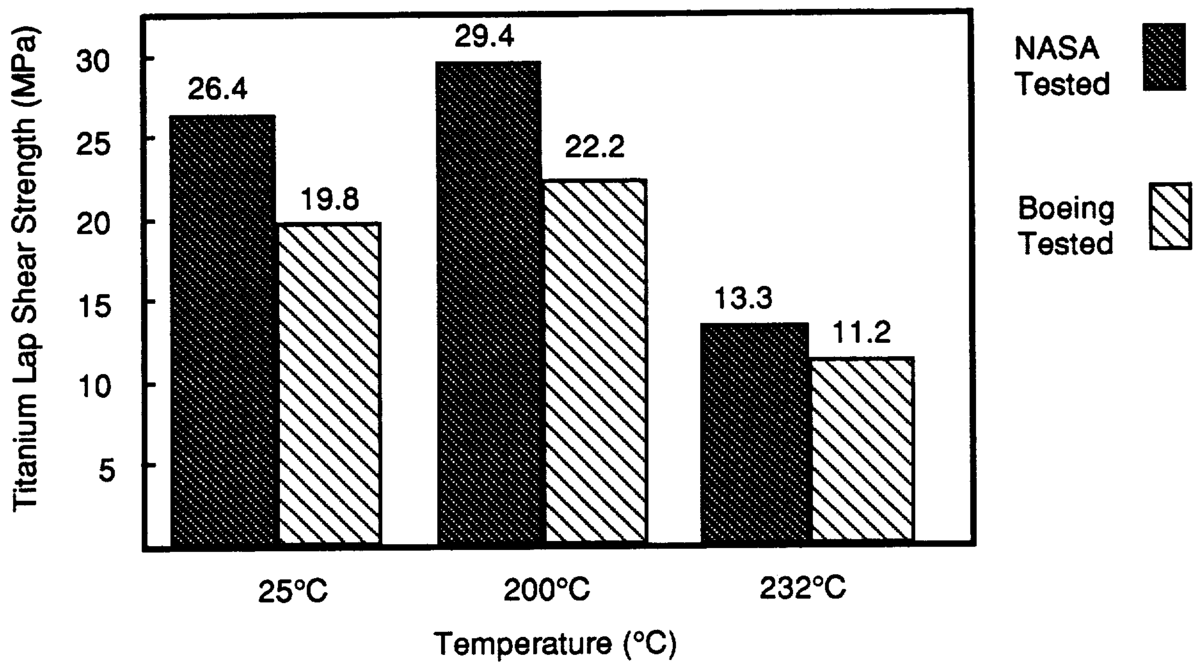


Figure 17. Comparison of Lap Shear Specimens Prepared at Boeing and Bonded at NASA. Tested at NASA and Boeing. All specimens post treated at 316°C for 100 hours.

performed at 375°C (707°F) and 1.38 MPa (200 psi) for 0.5 hour and the specimens were then heat treated at 308°C for 16 hours. The specimens were tested at Boeing and the lap shear strengths compared to values obtained at NASA (Table IX). Individual lap shear strengths are listed in Table X. In order to fit in the Boeing Instron test machine grips, 3.2 cm (1.25 inches) had to be trimmed from each end of the bonded lap shear specimen. Since the section removed was behind the grip pin holes, the lap shear specimen test results should not have been affected.

Table IX. Comparison of LARC-CPI Titanium Lap Shear Strengths Obtained at NASA and Boeing; Specimens Prepared at NASA

Test Temperature	NASA*	Boeing**
Ambient	34.0 MPa (4930 psi)	29.9 MPa (4330 psi)
177°C (350°F)	33.2 MPa (4820 psi)	27.1 MPa (3930 psi)
200°C (392°F)	28.1 MPa (4080 psi)	20.1 MPa (2910 psi)

*From letter of Paul M. Hergenrother dated January 31, 1990
**Average of two specimens.

Table X. LARC-CPI Lap Shear Strengths, Tested at Boeing

Specimen	Temp	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Surface
N-535	AMB	0.152 (0.006)	31.4 (4550)	100% Cohesive
N-536	AMB	0.127 (0.005)	28.3 (4100)	100% Cohesive
N-537	177°C	0.152 (0.006)	27.2 (3940)	100% Cohesive
N-538	177°C	0.152 (0.006)	27.0 (3910)	100% Cohesive
N-539	200°C	0.152 (0.006)	19.4 (2810)	100% Cohesive
N-540	200°C	0.178 (0.007)	20.7 (3000)	100% Cohesive

AMB = Ambient Temperature

Comparing the results in Table IX and Figure 18, Boeing obtained lower lap shear strengths at ambient temperature by 4.14 MPa (600 psi), at 177°C by 6.14 MPa (890 psi), and at 200°C by 8.07 MPa (1170 psi), or percentage differences of 12%, 18%, and 29% respectively. The test procedure or the setup of the test specimen in the test machine would have been the most likely causes of these differences. The test procedure and an analysis of the test specimens are discussed in Section 2.1.5.e.

2.5.1.c) Test Results from Final Batch of LARC-CPI

A 410 gram sample of polyamic acid solution of LARC-CPI in N,N-dimethylacetamide (30 percent solids, inherent viscosity of 0.53 dL/g, sample no. SH-69-82-20) was received from NASA which had a lower molecular weight than the previous LARC-CPI resin solutions used for adhesive bonding. Improved resin flow was expected from the lower molecular weight polymer. An adhesive tape nominally 13 mils thick was prepared from a portion of this resin solution.

Titanium lap shear specimens were bonded and tested at ambient temperature both before and after heat treatment at 316°C (600°F).

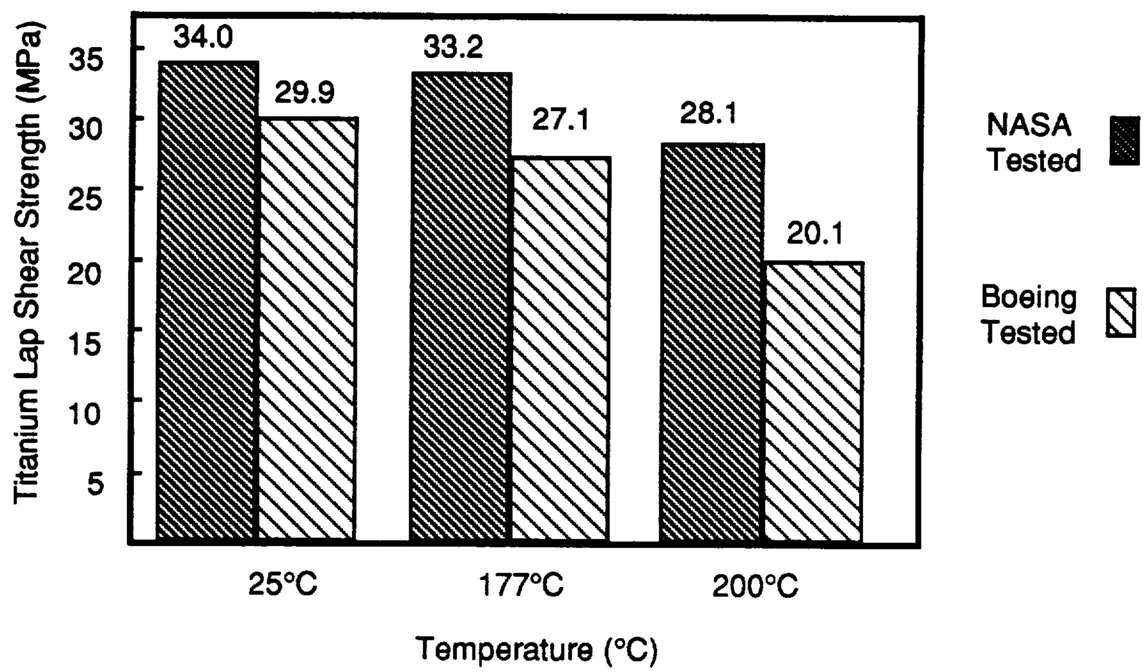


Figure 18. Comparison of LARC-CPI Titanium Lap Shear Strengths. Specimens prepared and bonded at NASA, and tested at NASA and Boeing (Table IX).

The lap shear strengths were low and no further testing was conducted at elevated temperature.

Essentially the same procedure (Figure 1) was used to prepare adhesive tape using a drying temperature of 250°C (480°F). The glass scrim was primed twice and dried and then 6 coatings were applied per side; the tape was dried after each coat. With 12 hours of additional drying time at 250°C the tape volatile content was 0.58 percent. The degree of flow of the tape was 35.6 wt% at 400°C and 24.9 wt% at 375°C under an applied pressure of 1.38 MPa (200 psi) for 15 minutes. Since lap shear strengths were highest when a high degree of resin flow occurred in the bondline, 400°C was selected for the bonding temperature rather than 375°C.

Titanium lap shear specimens were bonded at 400°C and 1.38 MPa for 15 minutes in the autoclave. A total of six sets of five specimens were bonded. Three sets were then heat treated for 100 hours at 316°C (600°F) in an air circulating oven. Two sets were tested at ambient temperature (Table XI). The average lap shear strength (Table XI) was 24.6 MPa (3570 psi) at ambient temperature. After heat treatment at 316°C the lap shear strength dropped to 16.6 MPa (2400 psi). The failure mode of these test specimens was about 70% cohesive. The lap shear strengths obtained by NASA using this same batch of resin solution and bonding conditions (with the exception of a bonding temperature of 375°C) for two specimens tested at ambient temperature were 33.5 MPa (4860 psi) and 26.4 MPa (3830 psi) . The failure mode of the NASA specimens was 100 percent cohesive.

As in previous results at Boeing with the LARC-CPI adhesive a drop in lap shear strength at ambient temperature after heat treatment at 316°C was observed. After heat treatment, adhesive failure appears to occur between the primer and the titanium conversion coating. Without heat treatment, adhesive failure occurs between the adhesive and primer.

These lap shear strength results were much lower than obtained with earlier batches of LARC-CPI (34.5 to 44.8 MPa, or 5000 to 6500 psi). The lower strengths may result from the lower molecular weight of the resin as compared to the previous batches of resin used in bonding. However, NASA obtained respectable strength (33.5 MPa at room temperature) with the lower molecular weight version of LARC-CPI.

Table XI. LARC-CPI Titanium Lap Shear Strengths - Individual Values

No heat treatment at 316°C; tested at ambient:

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
12A-1	0.102 (0.004)	24.1 (3490)	70% Co
12A-2	0.127 (0.005)	23.7 (3440)	70% Co
12A-3	0.178 (0.007)	24.4 (3540)	70% Co
12A-4	0.127 (0.005)	25.1 (3640)	70% Co
12A-5	0.152 (0.006)	25.9 (3760)	70% Co
	Average Std. Deviation COV	24.6 (3570) 0.883 (128) 0.04	

Heat Treatment at 316°C for 100 Hours and Tested at Ambient:

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
12B-1	0.127 (0.005)	17.7 (2560)	70% Co
12B-2	0.127 (0.005)	17.9 (2590)	70% Co
12B-3	0.127 (0.005)	13.8 (2000)	60% Co
12B-4	0.127 (0.005)	18.3 (2660)	60% Co
12B-5	0.152 (0.006)	15.2 (2200)	50% Co
	Average Std. Deviation COV	16.6 (2400) 1.98 (287) 0.12	

LARC-CPI resin solution batch number SH 69-82-20. Tape drying temperature: 250°C; volatiles content: 0.58 percent; degree of flow: 35.6 weight percent at 400°C. Co = Cohesive.

2.5.1.d) Bondline Surface Analysis

Surface analysis performed with Boeing funding revealed no differences in the fracture surfaces of titanium lap shear specimens (prepared from a Boeing LARC-CPI adhesive tape) from both NASA and Boeing. The analysis was performed by electron spectroscopy for chemical analysis (ESCA) and by auger electron spectroscopy (AES). As a follow-up to this investigation scanning electron micrographs (SEM) were prepared from sections of the lap shear specimens in which adhesive failure had occurred, and compared to Pasa-Jell 107 treated titanium. The surface analysis was performed with Boeing funds since this analysis fell outside the contract statement of work.

SEM micrographs of titanium treated with Pasa-Jell 107 and of titanium lap shear specimen fracture surfaces are shown in Appendix B. There appears to be no difference in the appearance of the treated titanium surface (Figure B1) after exposure to the bonding temperature of 400°C for 15 minutes (Figure B2). In both cases the surface appears rough and scaly. The exposed titanium surfaces have a similar rough and scaly appearance (Figures B3 - B6). The specimen prepared at NASA (Figure B3) did not appear any different from the specimens prepared at Boeing (Figures B4 - B6).

The SEM micrographs in Figure B7 showed a surface that had been grit blasted and treated with a standard hydrofluoric/nitric acid etchant for 90 seconds. The surface was relatively smooth compared to the Pasa-Jell treated specimens and may only have a scattering of titanium oxide over its surface. Surprisingly some of the photomicrographs of Pasa-Jell treated titanium appearing in some earlier NASA contract reports (Ref. 14) show a similar appearance.

In conclusion, the Pasa-Jell treated titanium appeared to have a suitably rough surface for mechanical bonding and both NASA and Boeing appeared to have achieved the same results in surface preparation.

2.1.5.e) Comparison of Testing Configurations and Analysis of Bondline Stresses

As far as could be determined, the NASA and Boeing testing procedures were essentially the same. Although the actual time the specimen was heated might vary by several minutes as long as the adhesive had reached the testing temperature the lap shear strength should remain about the same. Other possible variables included the test specimen dimensions and fixturing in the test machine. Boeing has been preparing and testing titanium lap shear specimens (Ref. 15) for many years, and the specimens being used were thought to be comparable to the NASA specimens.

Upon examining the bonded titanium specimens received from NASA, it was noted, however, that the pin hole-to-pin hole distance was 3.2 cm (1.25 inches) less for NASA specimens than for Boeing specimens. If the distance the adherends are supported in the grips was different for the specimens, could the peel stresses in the Boeing specimens be higher than in the NASA specimens?

Based on a set of lap shear test specimens bonded at NASA and sent to Boeing for testing, it appeared that differences in the gage lengths of NASA and Boeing specimens might be contributing to the differences in test results. A finite element analysis of the NASA and Boeing titanium lap shear specimens indicated that for the same test specimen gripping conditions the Boeing specimen will fail at approximately a 15 percent to 19 percent lower apparent stress level than the NASA specimen, ie. the Boeing specimen will have an equivalent stress state at a 15 percent to 19 percent lower applied load. It appears that differences in test specimen fixturing accounted for some of the discrepancy between NASA and Boeing test results although this was not the entire explanation.

A finite element model for adhesive bond lap shear specimens was available from a previous Boeing project to analyze the stresses in a lap shear joint for adhesively bonded thermoplastic composites. The model was developed by David J. Carbery of the BA&E Structures Technology Organization, who performed an analysis of the NASA and Boeing bonded titanium specimens. A minimal amount of effort was used for this analysis. However, the analysis was performed in enough detail to draw valid conclusions.

The mechanical properties that were used are listed in Table XII. Data for the DuPont K-III thermoplastic polyimide were used for the adhesive since all of the data needed were not available for LARC-CPI.

Table XII. Mechanical Properties for Finite Element Analysis of Lap Shear Joint

Quantity	Titanium 6Al-4V	AVIMID K-III
Young's Modulus (E)	110 GPa (16.0 Msi)	3.79 GPa (0.55 Msi)
Shear Modulus (G)	42.8 GPa (6.2 Msi)	1.37 GPa (0.198 Msi)
Poisson's Ratio (ν)	0.31	0.37

A two dimensional plane stress model representing a vertical section through the entire centerline of the specimen from grip pin to grip pin was used. The mesh in the lap bond area was fine enough to determine the general trends and indicate maximium stress values, and the mesh outside of the bond was coarser. A linear static analysis was performed.

The model was based on testing the NASA titanium specimens in the Boeing grips. In the NASA test procedure, however, the specimens are not gripped but are held only by a pin passing through a hole in each adherend. The shanks of the adherends are not gripped.

A diagram of the NASA and Boeing titanium lap shear specimens and griping conditions (Boeing test procedure) is shown in Figure 19. The supported length of the adherends was based on the grips used by Boeing for this testing. Each specimen in the model was fixed at one pin location and a 9,340 N (2100 lb_f) load was applied to the other pin location. The adherend sections within the grips were constrained from translation in the lateral (Y) direction, however, this is not the case for the NASA test procedure in which the specimens are attached only by pins.

The results of the analysis are sumarized in Table XIII and Figures 20 and 21. In Table XIII the peak stresses (axial, shear, and peel) were 15 to 19 percent higher for the Boeing lap specimen compared with the NASA specimen for the same load of 9,340 N (2100 lb_f). Application of the von Mises failure criterion showed a 17 percent difference in the equivalent stresses. Note that the deflections occurring in the Boeing specimen were about twice as high as in the NASA specimen (illustrated in Figures 20 and 21.)

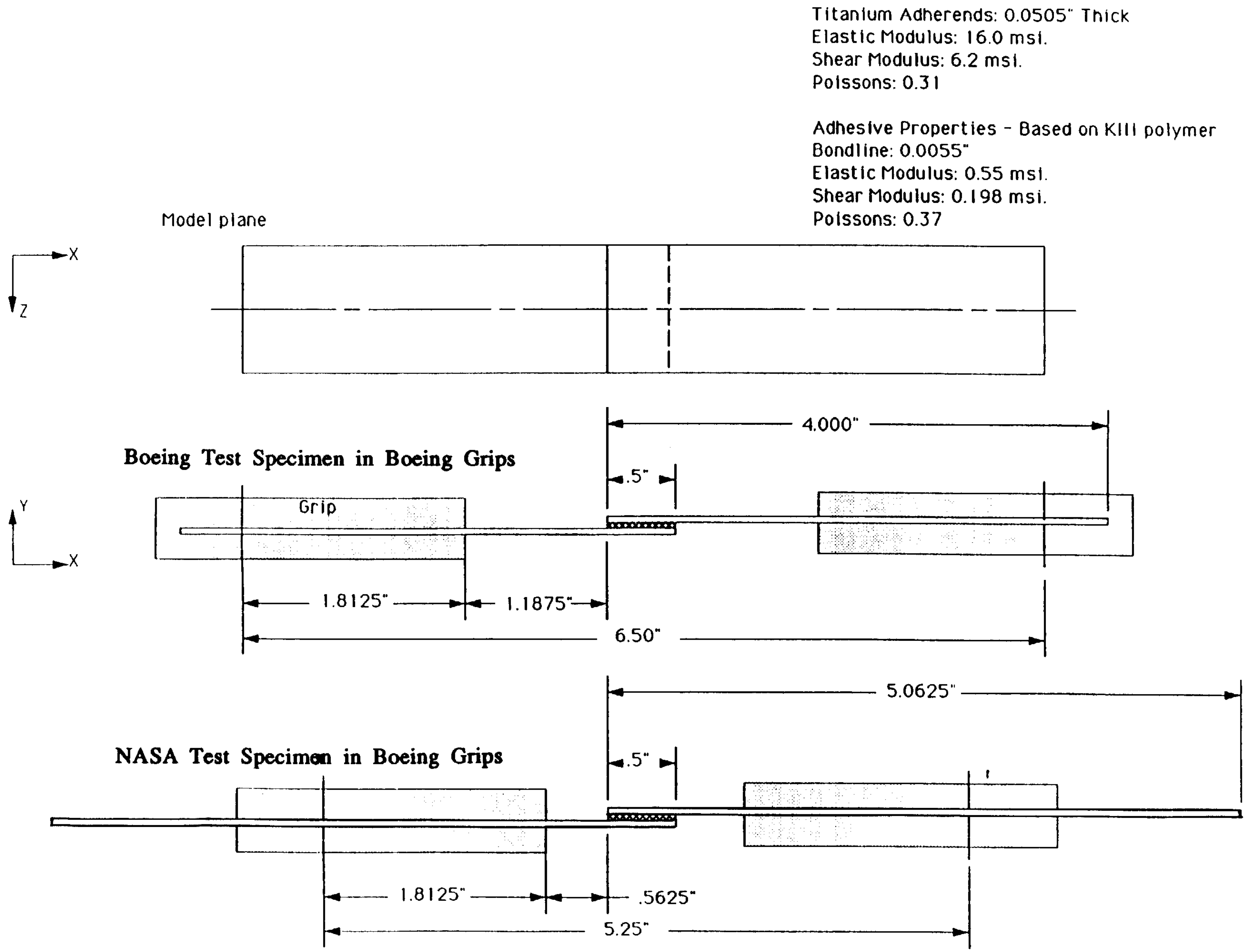


Figure 19. NASA and Boeing Titanium Lap Shear Specimen Dimensions and Grip Locations

Table XIII. Comparison of Stresses and Deflections in the NASA and Boeing Lap Shear Specimens for an Applied Load of 9,340 N (2100 lbf)

Quantity	NASA Specimen		Boeing Specimen		Percent Difference (maximum)
	Maximum	Minimum	Maximum	Minimum	
Overall Deflection	0.034633 cm	——	0.070777 cm	——	+ 104 %
Adhesive Deflection	0.023899 cm	——	0.051529 cm	——	+ 115 %
Peak Shear Stress (σ_{xy})	109.4 MPa	11.03 MPa	126.3 MPa	7.241 MPa	+ 15 %
Stress Ratio*		3.77		4.36	+ 15 %
Peak Peel Stress (σ_{xy})	218.3 MPa	-23.45 MPa	260.3 MPa	-30.83 MPa	+ 19 %
Peak Axial Stress (σ_x)	79.45 MPa	-3.8069 MPa	95.03 MPa	-6.193 MPa	+ 19 %
Peak von Mises Stress	275.2 MPa	19.72 MPa	323.4 MPa	13.45 MPa	+ 17 %

*Ratio of peak shear stress to calculated average shear stress.

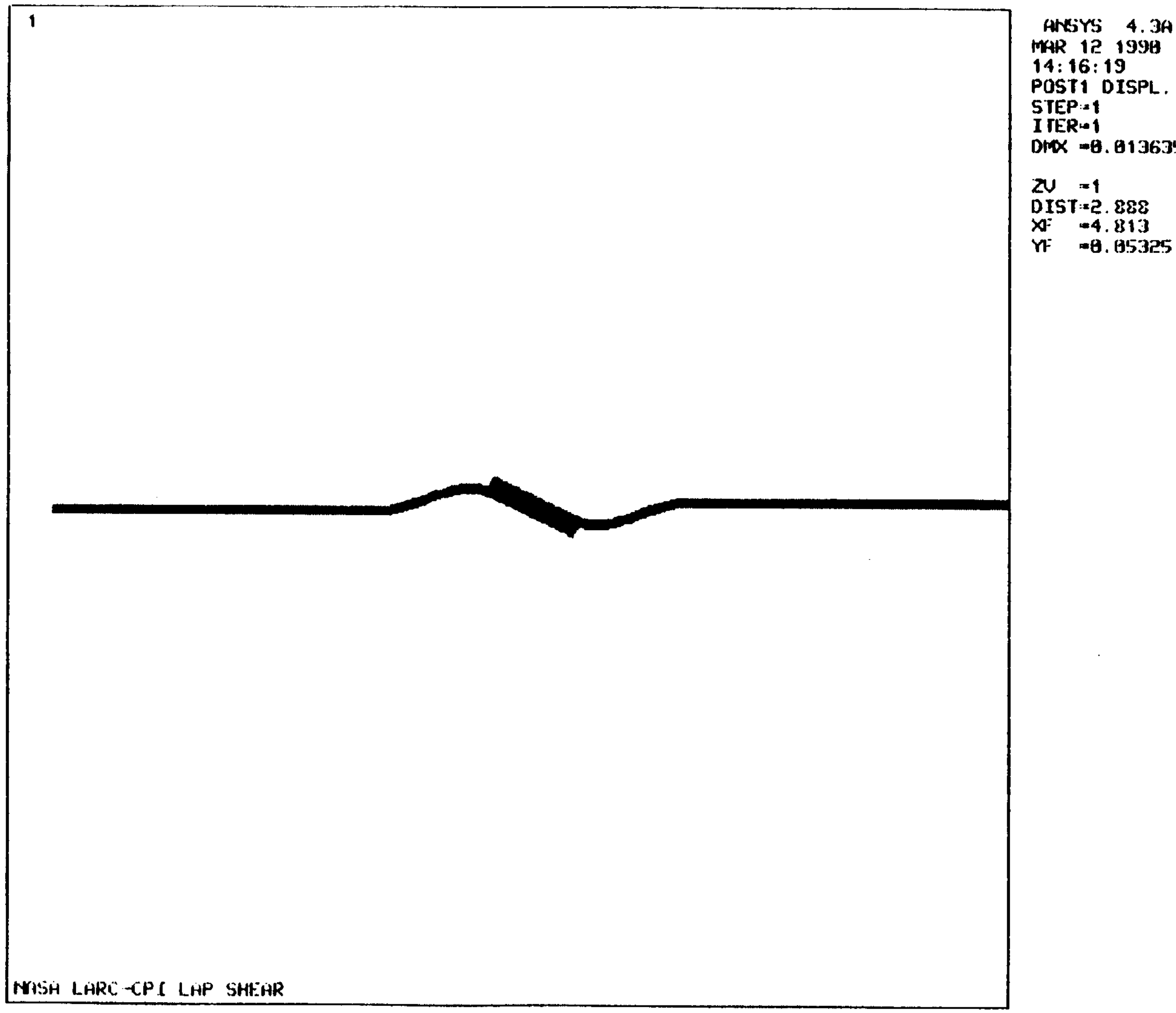


Figure 20. Deflection of NASA Titanium Lap Shear Specimen Under 9,340N (2,100-lbf) Load

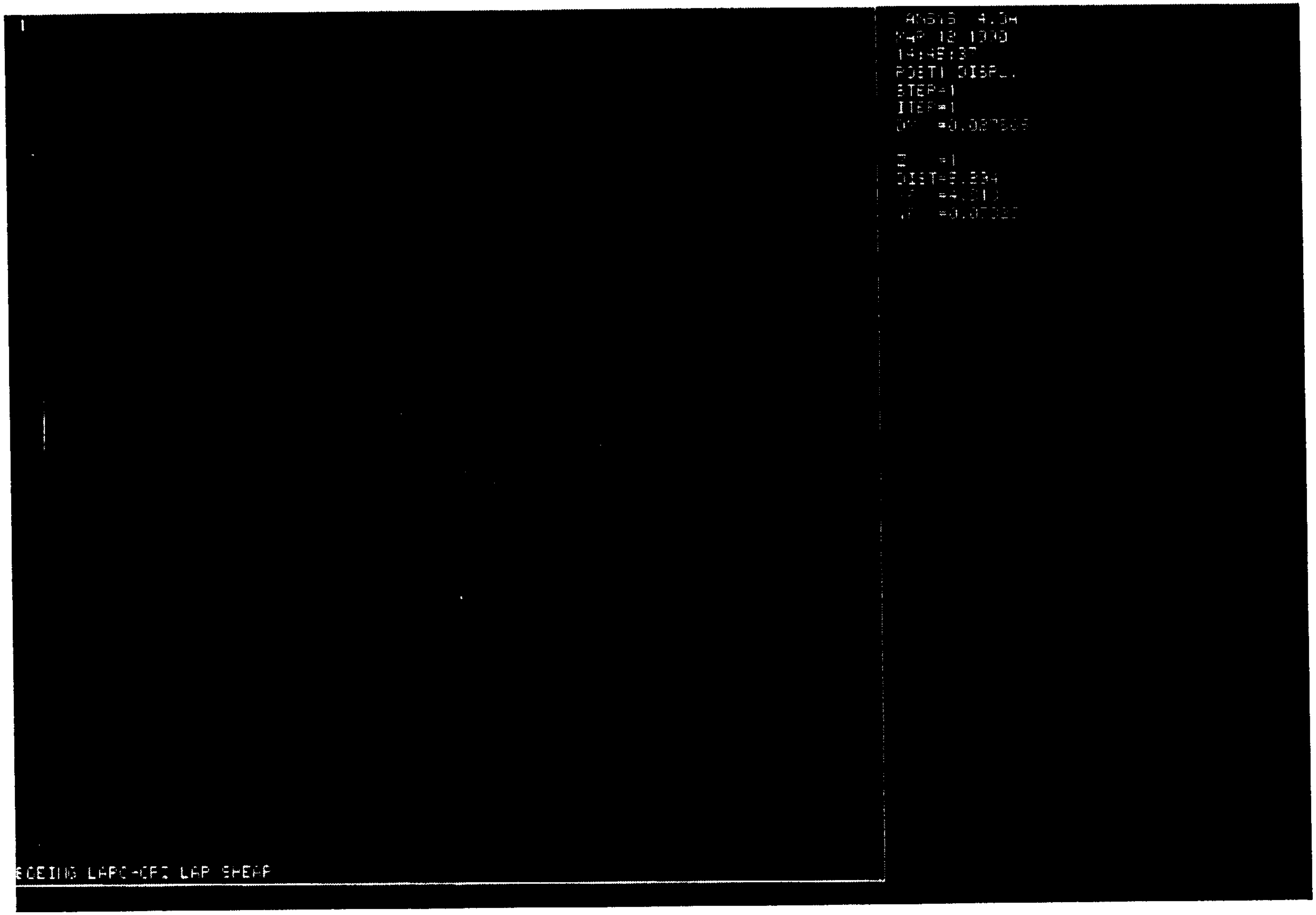


Figure 21. Deflection of Boeing Titanium Lap Shear Specimen Under 9,340N (2,100-lbf) Load

In conclusion the analysis indicated that for the same set of test machine grips, the greater length of the Boeing specimen extending from each grip (Figure 19) resulted in a doubling of the deflection, and a 15 percent to 19 percent increase in maximum stress levels at the bondline. The analysis predicted that the Boeing specimen will fail at a 15 percent to 19 percent lower apparent stress level than the NASA specimen (in the Boeing grips), ie. the Boeing specimen will have an equivalent stress state at a 15 percent to 19 percent lower applied load. The analysis of titanium lap shear specimens showed that even though titanium is a very stiff adherend, the manner in which the specimen is gripped can have a marked effect on the stresses that develop in the lap joint prior to bond failure.

ASTM specification D1002, "Standard Test Method for Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal)" specifies a distance of 6.35 cm (2.5 in) from the end of the lap to the end of the jaws; neither the NASA nor Boeing test specimens conform to this requirement with the Boeing grips. No jaws are used to constrain the adherends in the NASA test procedure. The Boeing grips and test specimens were standard Boeing test equipment and have been used in previous Boeing programs.

To provide further confirmation that a significant portion of the differences in NASA and Boeing lap shear test results were due to differences in test specimen gripping, modified specimens were tested. Autoclave bonded titanium lap shear coupons were tested with the same grip locations as the NASA specimens by drilling additional pin clearance holes.

Titanium lap shear specimens were tested with pin grips as used by NASA. The results were compared to earlier test results using wedge grips. A comparison of the NASA and Boeing lap shear specimen grip configurations is shown in Figure 19.

The titanium lap shear specimens were taken from specimens that had been bonded previously. The adhesive tape volatile content was 0.58 weight percent and the degree of flow was 35.6 weight percent at 400°C (applied pressure of 1.38 MPa (200 psi) for 15 minutes). The lap shear specimens were bonded at 400°C and 1.38 MPa for 15 minutes in an autoclave, and three of the six sets were then heat treated for 100 hours at 316°C (600°F) in an air circulating oven. At ambient temperature the average lap shear strength was 24.6 MPa (3570 psi) which dropped to 16.6 MPa (2400 psi) after heat treatment. The failure mode of these specimens was about 70 percent cohesive.

The lap shear strengths measured on the untreated and heat treated (100 hrs at 316°C) specimens using the pin grips were somewhat lower than obtained previously (Table XIV as compared to Table XI). On untreated lap shear specimens, a lap shear strength of 21.5 MPa (3120 psi) was measured using the pin grip arrangement compared with 24.6 MPa (3570 psi) using the wedge grips. After heat treatment for 100 hours at 316°C (600°F) the respective shear strengths measured 16.6 MPa (2410 psi) and 16.6 MPa (2400 psi). Although peel stresses would be higher in the unsupported specimens in the pin grips, there does not appear to be a significant difference in the results obtained with the two types of grip assemblies. To be consistent, the grips used and the pin-to-bondline distance should be the same in all specimens.

An interesting observation was that an adhesive failure surface seemed to predominate in the lap shear specimens tested with the pin grips. This might have resulted from higher peel stresses although the average shear strengths of the two sets of specimens appeared to be comparable.

Test specimen fixturing did not account for all of the discrepancies between NASA and Boeing test results. Test data (Table XV) show average strengths far below the results reported by NASA for specimens prepared from the same batch of resin. The most significant difference between our tape and NASA's is the volatiles content (0.58 versus 2.8%). When specimens were bonded earlier in the program with higher volatiles content tape, the resin turned very dark and low bond strengths and adhesive failures resulted.

Discussions with NASA personnel also revealed that a problem occurred with the use of an area compensator on one of their test machines causing strengths to be about 20 percent higher than the actual values. The combined factors of test specimen griping and incorrect use of a test machine area compensator probably accounts for some of the differences between the NASA and Boeing titanium lap shear test results. However, it is not known how many of the specimens tested at NASA were influenced by the compensator problem.

Table XIV. 6Al-4V Titanium Lap Shear Strengths Obtained Using Pin Grip Testing Arrangement - LARC-CPI Adhesive. All testing was performed at ambient temperature.

Specimen No. (no heat treatment)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Surface
12A-6	0.178 (0.007)	21.0 (3040)	50% Cohesive
12A-7	0.127 (0.005)	19.1 (2770)	40% Cohesive
12A-8	0.127 (0.005)	23.4 (3400)	40% Cohesive
12A-9	0.127 (0.005)	21.4 (3100)	50% Cohesive
12A-10	0.152 (0.006)	22.6 (3270)	50% Cohesive
Average Standard Deviation Coefficient of Variation		21.5 (3120) 1.66 (240) 0.08	
(heat treatment, 100 hrs @ 316°C)			
12B-6	0.127 (0.005)	15.0 (2170)	80% Adhesive (dark resin)
12B-7	0.178 (0.007)	18.3 (2660)	60% Cohesive
12B-8	0.152 (0.006)	17.7 (2560)	70% Adhesive
12B-9	0.152 (0.006)	17.1 (2480)	60% Adhesive
12B-10	0.152 (0.006)	15.2 (2200)	80% Adhesive
Average Standard Deviation Coefficient of Variation		16.6 (2410) 1.51 (219) 0.09	

Table XV. Comparison of NASA and Boeing LARC-CPI Bonded Titanium Lap Shear Specimens

Postcure Condition	NASA* Shear Stress MPa (psi)	Failure	Boeing** Shear Stress MPa (psi)	Failure
None	33.5 (4860)	100% Coh	24.6 (3570)	70% Coh
316°C, 100 hrs	26.4 (3830)	80% Adh	16.6 (2400)	60% Coh

*Letter from Paul M. Hergenrother dated September 14, 1989
**January, 1990 Progress Report (Table XI). Average of 5 specimens.

2.2 LARC-TPI Titanium Bonding

2.2.1 Resin Composition and Properties

LARC-TPI was evaluated for titanium honeycomb core bonding. Adhesive tapes were prepared from a slurry of fully imidized LARC-TPI powder and a polyamic acid solution in diglyme. Pasa-Jell 107 treated titanium honeycomb core (6.4 mm, or 0.25 inch cell size) was primed with dilute resin solution and then coated with the slurry. The tape and honeycomb were press or autoclave bonded with Pasa-Jell 107 treated and primed titanium face skins (0.76 mm thick).

Uniformly thick coatings on each side of the tape were difficult to produce. Cracking and peeling of the adhesive would often occur on the tape. Coating both sides of the tape for each drying cycle and predrying to flash off excess solvent at lower temperatures improved tape uniformity.

Producing a uniform coating of the slurry on the honeycomb core was also challenging. A uniform adhesive coating along the edges of the core was needed that would reticulate and form fillets between the honeycomb core and face sheets. If the coating was too thick excessive volatiles could be trapped in the resin and if too thin adequate fillets might not form, both conditions resulting in lower bond strengths.

The LARC-TPI resin solution used to produce all prepreg slurries was Durimid 100 manufactured by the Rogers Corporation. Four different imidized LARC-TPI powders were used to bond flatwise tensile and titanium lap shear specimens. Although very good flatwise tensile strengths were obtained from some LARC-TPI powders, lot to lot variation of the powders was evident. Recent efforts at both NASA and licensed producers of LARC-TPI resins have been directed at producing powders with improved, more consistent flow characteristics (Ref. 12).

Both the LARC-TPI resin solution and powder were obtained from the Rogers Corporation, Rogers, CT. The LARC-TPI resin solution was a commercially available product, trade named DURIMID 100, and contained 30 weight percent solids in diglyme (lot no. 354, inherent viscosity of 0.5 dl/g at 25°C). The Rogers LARC-TPI powder (lot no. 488) is tradenamed DURIMID 500P and had a 5 to 10 micron particle size distribution.

A second batch of LARC-TPI powder (lot #92-709) produced by Mitsui-Toatsu was obtained from Dr. Richard Moulton at Hexcel. Powder from this lot was also used in the prepreg produced for composites evaluation. The Mitsui-Toatsu powder had a lower molecular weight than the Rogers powder, and had a fairly sharp melting transition at about 280°C. The Rogers powder did not have a clearly visible melting transition when observed in a melting point apparatus. Agglomeration of the Rogers powder began to occur at around 270°C. Differential scanning calorimetry (DSC) traces of both powders had melting endotherms at 300°C (570°F) for the Rogers powder and 280°C (540°F) for the Mitsui Powder.

A small sample (about 20 grams) of a different Mitsui-Toatsu LARC-TPI powder, designated the 1500 series, was subsequently received from NASA-Langley. This powder had a melting temperature of around 320°C and was reported to have improved flow properties.

2.2.2 Adhesive Tape Preparation

The LARC-TPI polyamic acid solution (30% solids) in diglyme was diluted with diglyme, and LARC-TPI powder was added to produce a slurry of 19% solids by weight. A primer was also prepared by a 3:1 dilution of the polyamic acid solution with diglyme.

An adhesive tape was prepared from style 112 E-glass scrim with an A1100 finish in a similar manner as for the LARC-CPI adhesives. Dried Style 112 E-glass scrim was primed with three coats of a 3:1 dilution of the LARC-TPI polyamic acid solution in diglyme. The powder was mixed with the LARC-TPI diglyme solution, which had been diluted with diglyme from 30 percent to 19 percent solids. Four coats of the LARC-TPI slurry were applied with a plastic sweep to produce a tape with a thickness of 0.33 to 0.36 mm (0.013 to 0.014 in). A 200°C drying cycle (Figure 22) was used to dry each coating including primer coatings.

Prior to drying the solvent was allowed to flash off in air for a period of at least 30 minutes. The coatings with the Mitsui-Toatsu powder were predried in a circulating air oven at 50°C for thirty minutes to avoid blistering of the powder coating.

The adhesive tape produced with the Rogers' LARC-TPI powder had a volatile content of 2.3 percent. Resin flow tests were performed at 1.38 and 6.90 MPa (200 and 1000 psi), at 300, 330, 340, and 400°C for 30 minutes. However, no flow occurred outside the circumference of the specimen disc although the resin did melt. The resin did flow somewhat under 6.90 MPa but the flow was confined within the specimen. The adhesive tape produced with the Mitsui-Toatsu powder had a volatiles content of 4.5 percent and a flow of 10.2 percent which were reduced to 2.4 percent and 7.0 percent respectively after drying for an additional 8 hours.

A small adhesive tape (15.2 cm by 15.2 cm) was prepared from the sample of 1500 series LARC-TPI powder received from NASA-Langley. This is a high flow version of the LARC-TPI powder, manufactured by the Mitsui-Toatsu company. The adhesive tape was prepared with a 40 percent solids slurry of the powder. The slurry was produced from a 30 percent solids solution of the LARC-TPI polyamide acid in diglyme and additional diglyme solvent. Style 112 E-glass fabric with an A1100 finish was primed with three coats of a 20 percent solids slurry followed by 4 coats of the 40 percent solids slurry. The 4 coats were applied two at a time (top and bottom of the tape) and allowed to dry at room temperature for 30 minutes followed by drying for 30 minutes at 50°C (120°F). The primer coats and the 4 coats were dried at 200°C (390°F) according

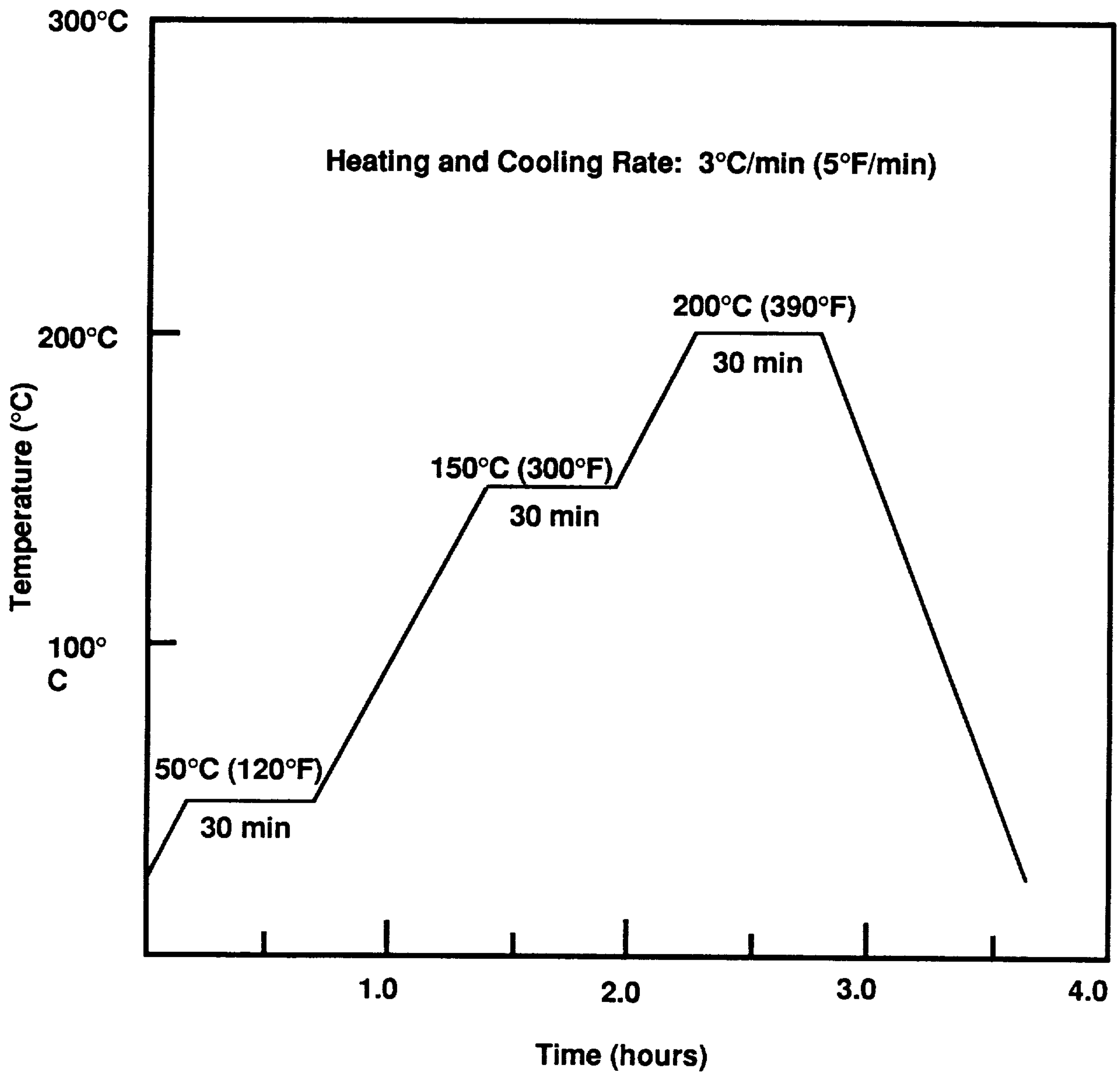


Figure 22. Drying Cycle for LARC-TPI Adhesive Tape

to the cycle shown in Figure 22. Some cracking of the resin occurred during drying of the tape. The tape had a volatiles content of 1.75 weight percent, and a thickness averaging 0.76 mm (30 mils).

2.2.3 Titanium Bonding and Mechanical Test Results

Several 6Al-4V titanium lap shear specimens were bonded with the adhesive tape prepared from the Rogers LARC-TPI powder to evaluate the bonding characteristics of this adhesive before bonding titanium honeycomb core.

Two 6Al-4V titanium lap shear specimens were prepared from the tape. The titanium was treated with Pasa-Jell 107 and primed with the dilute resin solution. A bonding pressure of 6.90 MPa (1000 psi) at 650°F for 30 minutes was used. The specimens had bondline thicknesses of 0.203 mm (0.008 in) and lap shear strengths of 22.5 and 28.6 MPa (3260 and 4150 psi). Ten additional specimens were bonded at 12.4 and 6.9 MPa (2000 and 1000 psi) and had average strengths of 28.0 and 26.5 MPa (4054 and 3840 psi) respectively (Appendix A, Table A7). Lower bonding pressures should have been used which would be appropriate for core sandwich bonding. However, the high temperature autoclave was down for repairs, and the low pressure hydraulic system of our smallest platen press malfunctioned.

To produce titanium sandwich test specimens powder slurries were applied to both sides of the honeycomb core by dip coating and drying each coating using the same drying cycle. Two coatings of the slurry were put on each side of the honeycomb core. The titanium honeycomb core had been treated with Pasa-Jell 107 using the process developed by NASA-Langley. Two coats of the primer were applied to the honeycomb and dried prior to the dip coating.

The titanium honeycomb core [6.4 mm (0.25 in. cell size)] and 0.76 mm (0.030 in.) thick face sheets were bonded in a press in a Kapton envelop bag under a nitrogen atmosphere. One layer of adhesive tape was placed between the honeycomb core and each face sheet. A bonding pressure of 0.31 MPa (45 psi) was applied and the part was held at 340°C (650°F) for thirty minutes.

Titanium honeycomb core specimens were first fabricated from the Rogers and Mitsui versions of LARC-TPI powder and tested in flatwise tension. The Mitsui Toatsu powder produced a much better bond between 6Al-4V titanium honeycomb core and face sheets than the Rogers' powder, probably due to the better flow characteristics of the Mitsui powder. The Rogers LARC-TPI powder did not have sufficient flow to produce adequate bonds at lower pressures or to be a suitable adhesive for bonding honeycomb core at lower pressures (0.31 MPa, or 45 psi).

The sandwich assembly bonded with Rogers LARC-TPI powder debonded during machining of the test specimen. A flatwise tensile specimen was machined from the sandwich bonded with the Mitsui-Toatsu powder, which had a tensile strength of 2.84 MPa (413 psi). Flatwise tensile strengths of 4.07 and 4.34 MPa (590 psi and 630 psi) were obtained on a prior NASA-Langley adhesives contract (Ref. 14) using a different version of the Mitsui-Toatsu LARC-TPI powder.

The Rogers LARC-TPI powder did not exhibit sufficient flow for fillet formation in honeycomb bonding. If the Rogers powder did have better flow characteristics it would be expected to have higher bond strengths because of its higher molecular weight. It might be possible to improve the flow of the Rogers powder by blending it with the Mitsui-Toatsu powder, however, blending was not tried.

The improved flow 1500 series LARC-TPI powder was then used to bond a titanium honeycomb section to titanium face sheets. The 6Al-4V titanium honeycomb core and face sheets were treated with Pasa-Jell 107 and primed with two coats of a 20 percent solids slurry and dried per the schedule in Figure 22. Fillets of the LARC-TPI powder were built up on the honeycomb core by allowing the core to stand in a 40 percent solids slurry and slowly dry. After about 5 days a sufficient resin fillet had built up on the core which was then dried at 200°C per Figure 22.

The coated core and titanium face sheets were bonded with the adhesive tape at 343°C (650°F) and 0.31 MPa (45 psi) for thirty minutes. A 5.0 cm by 5.0 cm flatwise tensile test specimen was cut from the bonded sandwich and tested at ambient temperature. A flatwise tensile strength of 4.83 MPa (700 psi) was obtained which compares very well with values of 4.34 MPa (630 psi) and 4.07 MPa (590 psi) obtained with LARC-TPI powder in a previous NASA contract (Ref. 14). Flatwise tensile strength minimum requirement values (aluminum honeycomb core, 9.53 mm cell size) listed in the Boeing Material Specification BMS 5-104D (Ref. 16) for aircraft structural 350°F cure adhesives is 3.10 MPa (450 psi) at room temperature and 1.38 MPa (200 psi) at 177°C (350°F). Core with a larger cell size would be expected to have lower flatwise tensile strengths than core with a smaller cell size.

Additional titanium honeycomb sandwich specimens were then bonded using Mitsui 1500 series powder (lot #2410, grade 2347) and tested at ambient temperature and 232°C. These specimens were prepared using Boeing funds. Both the titanium face sheets and core were treated with Pasa-Jell 107 prior to priming for bonding. The titanium face sheets were bonded using adhesive prepared from a slurry of the 1500 series LARC-TPI powder as described in previous progress reports. The titanium core was roller coated with a 1:2 slurry by weight of the 1500 series LARC-TPI powder in LARC-TPI polyamide acid resin solution in diglyme. After priming and drying the core twice, two coatings of the LARC-TPI slurry were applied and dried at 200°C.

The flatwise tension specimen test results are listed in Table XVI. Specimen no. 1 was prepared individually as described previously. The test results at both ambient temperature and 232°C were very satisfactory. LARC-TPI powder slurries had flatwise tensile strengths (average of two specimens) of 4.59 MPa (666 psi) and 2.88 MPa (417 psi) at ambient temperature and 232°C respectively. Photographs of specimens 2 and 4 are shown in Figure 23.

Additional titanium honeycomb sandwich specimens were bonded using a different lot of the 1500 series powder (lot #58-704) and tested at ambient temperature. Both the titanium face sheets and core were treated with Pasa-Jell 107 and then primed. The titanium face sheets were bonded using adhesive prepared from a slurry of the 1500 series LARC-TPI powder as described previously. The titanium core was roller coated with a 1:2 slurry by weight of the 1500 series LARC-TPI powder in LARC-TPI polyamide acid resin solution in diglyme. After priming and drying the core twice, two coatings of the LARC-TPI slurry were applied and dried at 200°C.

Table XVI. Titanium Core Flatwise Tensile Strength Test Results - Mitsui 1500 Series LARC-TPI Powder

Specimen No.	Test Temp.	Compressive Strength
1	Ambient	4.83 MPa (700 psi)
2	Ambient	4.35 (631)
Average		4.59 (666)
3	232°C (450°F)	3.07 (445)
4	232°C (450°F)	2.68 (388)
Average		2.88 (417)

The flatwise tension specimen test results are listed in Table XVII. The test results may have been low because the panels, which were bonded in the autoclave, did not reach the bonding temperature of 343°C (650°F); instead the temperature only reached 338°C (640°F) due to a temperature controller problem. The bonding was to be performed at 343°C for 30 minutes at 0.31 MPa (45 psi). The resin in the specimens was a very light tan color, whereas the resin in previous specimens turned a very dark brown color after bonding.

Table XVII. Titanium Core Flatwise Tensile Strength Test Results - Mitsui 1500 Series LARC-TPI Powder (Lot #58-704)

Specimen No.	Test Temp.	Flatwise Tensile Strength
TPI-101	Ambient	2.68 MPa (389 psi)
TPI-102	Ambient	1.72 (250)
TPI-103	Ambient	2.48 (360)
	Average	2.30 (333)

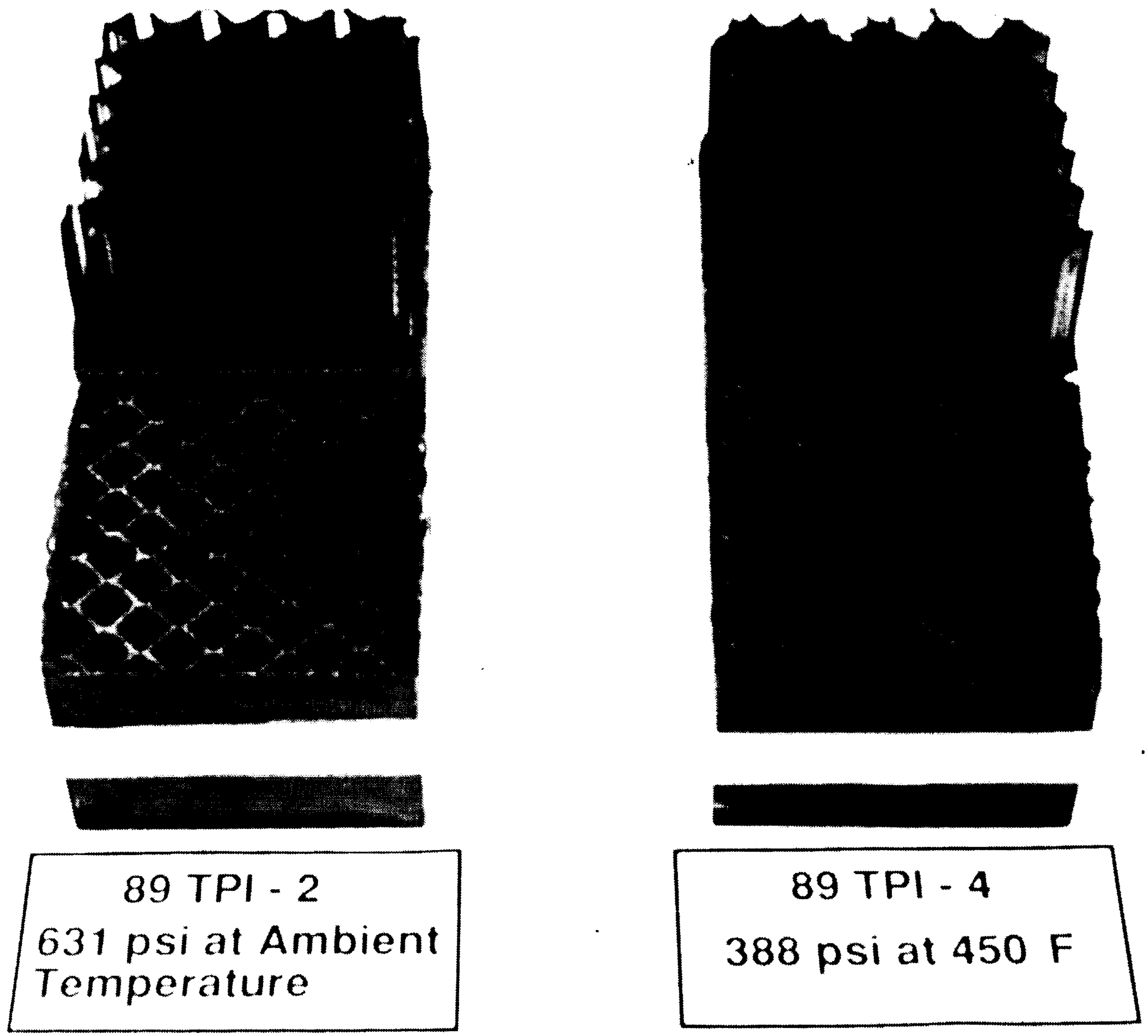


Figure 23. Titanium Honeycomb Core Bonded With LaRC-TPI, With Flatwise Tensile Strengths Noted

Another sandwich panel was bonded with the LARC-TPI slurry and tape at 343°C and three additional flatwise tensile specimens were tested at ambient and three at 232°C. The resin in this panel still looked very light in color after bonding. This may be due to some characteristic of this lot of powder (lot #58-704). The specimens tested previously were prepared from adhesive tape produced with LARC-TPI powder lot #2410. Since the high temperature autoclave was down for repairs this sandwich panel was bonded in a heated platen press using the same bonding conditions.

Additional titanium honeycomb sandwich specimens were bonded using the 1500 series powder (lot #58-704) and tested at ambient temperature. The titanium core was roller coated with a 1:2 slurry by weight of the 1500 series LARC-TPI powder in LARC-TPI polyamide acid resin solution in diglyme. After priming and drying the core twice, two coatings of the LARC-TPI slurry were applied and dried at 200°C. The bonding was accomplished by holding the sandwich at 343°C for 30 minutes at 0.31 MPa in a heated platen press.

The flatwise tension specimen test results are listed in Table XVIII.

Table XVIII. Titanium Core Flatwise Tensile Strength Test Results

Specimen No.	Test Temp.	Flatwise Tensile Strength MPa (psi)
789-1	Ambient	1.11 (161)
789-2	Ambient	2.13 (309)
789-3	Ambient	2.39 (347)
	Average	1.88 (272)

The flatwise tensile strengths were probably low because the bonding temperature of 343°C (650°F) that was successfully used to bond the sandwich specimens from the lot #2410 powder was not high enough to achieve adequate flow of the lot #58-704 powder. Although fillet formation did occur it was not as complete and uniform as on the previous specimens that had higher flatwise tensile strengths. Discussions with Mr. Timothy Towell of NASA-Langley have indicated that higher process temperatures give better results with this powder in composites. At 370°C (700°F) the powder will have a very low melt viscosity and will remain amorphous on cool down. At 343°C (650°F) the powder will not have adequate melt flow and will recrystallize on cool down.

Differential scanning calorimetry (DSC) was performed on resin samples from specimen 789-2. The DSC traces showed a melting endotherm at 350°C (662°F), evidence that the resin crystallized on cool down.

3.0 Composites

3.1 LARC-TPI Composite Prepregs

Composite laminates were fabricated in both the press and autoclave from prepreg material produced by Dr. Richard Moulton of A. M. Technology Inc. The prepreg resin was a 1:1 slurry by weight of LARC-TPI powder in a polyimidesulfone ($PISO_2$)/diglyme solution, with 5 weight percent of a bisamideacid (BAA) (Ref. 17). The BAA and the semicrystalline LARC-TPI powder (lot no. 92-709) were added to improve the processability and resin flow of LARC-TPI composite matrices. The carbon fiber reinforcement was unsized Hercules AS4 which was treated just prior to prepregging with BAA to facilitate handling of the fibers.

Two rolls of prepreg totaling approximately 6.8 Kg (15 lbs) were received. The solvent content was lower than in previous batches, to minimize the splitting that occurs when working with the tape as the solvent evaporates. The tape was quite boardy. The tape was of moderate to high quality. There were some narrow axial areas along the tape where LARC-TPI powder had collected.

The prepreg tapes were characterized for volatiles and resin content (Table XIX).

Table XIX. $PISO_2$/LARC-TPI Prepreg Tape Physical Properties

Batch	Roll	Weight of Prepreg Kg (lbs)	Volatiles Content* (wt%)	Resin Content (wt%)
DT-623	02	3.5 (7.8)	12.1	38.5
			11.9	38.8
			11.3	39.1
Average			11.8	38.6
DT-623	03	3.4 (7.6)	12.0	39.6
			12.3	42.0
			12.5	41.7
Average			12.3	41.1

*Drying at 315°C (600°F) for 30 minutes.

3.2 Laminate Fabrication

Press consolidation was performed using a processing cycle (Figure 24) suggested by Dr. Norman Johnston of NASA-Langley. The prepreg tape was dried in an oven at 200°C (400°F) for one hour between layers of permeable Armalon. The prepreg was then cut to size and stacked in the layup, with two plies per layer to compensate for tape splitting. Crossply layups of 16 and 32 plies were consolidated to assess processability. Well consolidated 32 ply quasi-isotropic laminates were desired for compression-after-impact testing.

No ideal method of tacking together successive plies in the layup was found. Using a tacking iron gave adequate results, however, adhesion between the plies was minimal and the layups had to be handled carefully.

The layup was placed between two sheets of Kapton film coated with Frekote 700 release agent and consolidated in a press with heated platens using a picture frame tool. The resin flow that occurred during processing was minimal, but sufficient to produce good quality laminates.

Through-transmission ultrasonic (TTU) scans were produced of each laminate and selected TTU scans are reproduced in Appendix C. The TTU scans were produced using a signal voltage of 6 volts and a frequency of 5 MHz. Higher numbers on the scan plot indicate higher levels of signal attenuation. Numbers greater than 3 or 4 indicate significant porosity.

Autoclave processing was conducted using the processing cycle recommended by NASA (Figure 24). The major difference from press processing was that the prepreg was dried as part of the autoclave cycle (at 200°C for 30 minutes). Two autoclave runs were performed.

The five laminates produced in the first autoclave run are listed in Table XX (887B1 through 887B5). The layups were consolidated on a steel vacuum tool. Layups were placed between two layers of permeable Armalon (top and bottom), a layer of Kapton film (top and bottom), and boat cloth breather layers, all under a Kapton bag. The autoclave could not reach the setpoint pressure of 2.1 MPa (300) psi, reaching instead a maximum of 1.59 MPa (230 psi).

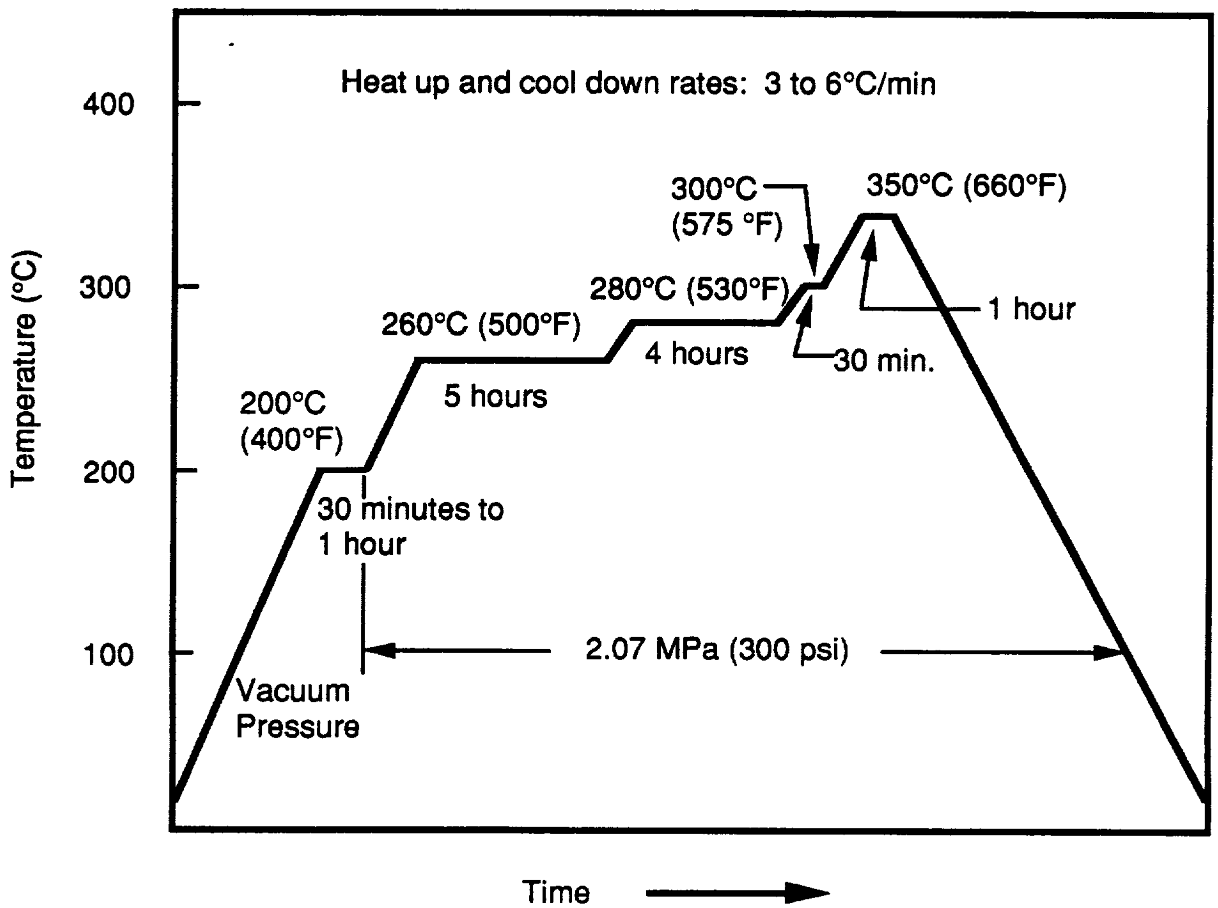


Figure 24. Process Cycle for Autoclave Consolidation of LARC-TPI Laminates

The TTU scans of the five laminates indicated that the thin, unidirectional laminates have acceptable consolidation. The laminate having a larger area (887B4) appears to have more porosity than the smaller twelve ply laminate (887B3), except for one area. The 32 ply crossply laminate (887B5) was poorly consolidated, and little resin flow was evident along the edges of the laminate. In general the autoclave consolidated laminates exhibited much less resin flow and poorer consolidation than the press consolidated laminates, probably because a lower autoclave pressure was used due to equipment malfunction.

For the second autoclave run, two layers of style 181 and two layers of style 120 glass fabric were added to the tool layup above and below the permeable Armalon which was next to the part. The process cycle in Figure 24 was modified also, increasing the dwell at 200°C (400°F) from 30 to 60 minutes, and removing the vacuum pressure immediately after the dwell and opening the vacuum lines to the atmosphere. The autoclave pressure reached the desired 2.07 MPa.

TTU scans of the three laminates (887C1 through 887C3) produced in the second autoclave run show some improvement in consolidation, particularly in the 2 ply laminate (887C3). The laminates also exhibited improved resin flow along their edges. The porosity in the 32 ply laminate was still unacceptable, however. The autoclave consolidated laminates were not as well consolidated as the press consolidated laminates (2.07 MPa consolidation pressure in both autoclave and press). The only major difference between the press and autoclave consolidation processes was the initial drying step in which the polyamic acid was mostly converted to polyimide, and the condensation byproducts and diglyme solvent were removed. Perhaps these volatiles were more effectively removed by drying the individual plies prior to layup and press consolidation, than was the case with the autoclave consolidated laminates which were only dried in the vacuum bagged layup.

Table XX. LARC-TPI Laminates Consolidated in Press and Autoclave

Laminate No.	Layup	Laminate Dimensions (cm)	Mechanical Testing
Press:			
886A1-DT623	$(0/90)_{4s}$	15.2 x 10.2	N/A
886A2-DT623	$(0/90)_{4s}$	15.2 x 10.2	N/A
886A3-DT623	$(0/90)_{8s}$	15.2 x 10.2	N/A
886A4-DT623	$(0/90)_{8s}$	15.2 x 10.2	N/A
Autoclave - 1st Run:			
887B1-DT623	$(0)_{10}$	15.2 x 30.5	0 Deg Compression
887B2-DT623	$(0)_{8}$	15.2 x 30.5	0 Deg Tension
887B3-DT623	$(0)_{12}$	15.2 x 30.5	N/A
887B4-DT623	$(0)_{12}$	30.5 x 30.5	90 Deg Tension
887B5-DT623	$(0/90)_{8s}$	15.2 x 15.2	N/A
Autoclave - 2nd Run:			
887C1-DT623	$(0)_{8}$	15.2 x 30.5	0 Deg Tension
887C2-DT623	$(0)_{12}$	30.5 x 30.5	90 Deg Tension
887C3-DT623	$(0/90)_{8s}$	15.2 x 15.2	N/A

A well consolidated 32 ply crossply laminate was fabricated in the press by holding the laminate under pressure for a long period of time. The press consolidation was interrupted after the 260°C dwell and the laminate left overnight in the press under pressure. Processing was completed the following day. A TTU scan (Figure C12) indicated that the laminate was very well consolidated.

Three larger (30.5 cm by 15.2 cm) unidirectional laminates that were 8, 10, and 20 plies thick and were press consolidated using the same process cycle had poor TTU scans. Additional drying time may be necessary for press consolidation of larger laminates.

Physical properties of the three laminates that were tested are listed in Table XXI. Fiber volume fractions were determined by acid digestion with sulfuric acid. Based on the TTU scans these three unidirectional laminates appeared to be the best quality laminates.

Table XXI. Laminate Physical Properties - PISO2/LARC-TPI Composites

Laminate	Fiber Volume (%)	Void Content (%)	Laminate Density (g/cm^3)
887B3-DT623:			
1	51.1	<1.0	1.57
2	50.7	<1.0	1.58
3	51.4	<1.0	1.60
Average	51.1	<1.0	1.58
887C1-DT623:			
1	53.8	<1.0	1.61
2	53.9	<1.0	1.62
Average	53.9	<1.0	1.62
887C2-DT623:			
1	53.5	<1.0	1.60
2	51.5	<1.0	1.59
3	52.4	<1.0	1.58
Average	52.5	<1.0	1.59

Specific gravities of LARC-TPI resin = 1.30 and AS-4 carbon fiber = 1.80.

3.3 Mechanical Test Results

Mechanical testing comprised tensile and compression testing at both ambient and elevated (177°C) temperature. The tensile test method used was ASTM D3039-76, "Standard Test Method for Tensile Properties of Fiber Resin Composites." A 177°C cure epoxy adhesive (FM-300) was used to bond the tabs on the elevated temperature test specimens. The tensile test results are listed in Table XXII. The 0 degree tensile specimens could not be tested at elevated temperature due to tab debond.

Compression testing was performed at ambient and elevated temperature on specimens from a 10 ply unidirectional laminate. The compression tests were conducted in accordance with ASTM D695 "Standard Test Method for Compressive Properties of Rigid Plastics", which had been modified by the Boeing Company for continuous fiber-reinforced composite laminates. Compression test results are listed in Table XXIII. Some tab debond occurred in the compression specimens and endbrooming failures occurred in many of the specimens.

Table XXII. Tensile Strength, Modulus, and Strain at Failure for AS4-PISO2/LARC-TPI Composites

Specimen	Strength MPa (ksi)	Modulus GPa (msi)	Ultimate Strain (%)
0 Degree*:			
1	1,270 (184)	138 (20.0)	0.92
2	1,100 (160)	127 (18.4)	0.87
3	1,440 (209)	112 (16.3)	1.29
4	1,270 (184)	131 (19.0)	0.97
5	1,350 (196)	120 (17.4)	1.13
Average	1,290 (187)	126 (18.3)	1.04
Std. Dev.	125 (18.1)	9.86 (1.47)	0.17
COV	0.10	0.08	0.17
90 Degree, Ambient:			
1	27.2 (3.94)	10.7 (1.55)	0.24
2	47.0 (6.82)	8.9 (1.29)	0.50
3	47.5 (6.89)	10.0 (1.45)	0.46
4	47.9 (6.94)	9.17 (1.33)	0.52
Average	42.4 (6.15)	9.72 (1.41)	0.43
90 Degree, @ 177°C:			
1	19.9 (2.88)	8.90 (1.29)	0.23
2	29.4 (4.26)	7.38 (1.07)	0.38
3	27.6 (4.00)	8.07 (1.17)	0.34
4	31.2 (4.53)	7.52 (1.09)	0.42
Average	27.0 (3.92)	8.00 (1.16)	0.34

* Normalized to a 60% fiber volume fraction.

Table XXIII. Compression Strength, Modulus, and Strain at Failure for AS4-$PISO_2$/LARC-TPI Composites

Specimen	Strength MPa (ksi)	Modulus GPa (msi)	Ultimate Strain (%)
Ambient:			
1	903 (131)	110 (16.0)	0.84
2	903 (131)	114 (16.6)	0.83
3	973 (141)	106 (15.4)	0.91
4	876 (127)	115 (16.7)	0.73
5	759 (110)	117 (17.0)	0.64
Average	883 (128)	112 (17.3)	0.79
Std. Dev.	77.9 (11.3)	4.47 (0.65)	0.11
COV	0.09	0.04	0.13
177°C:			
6	601 (87.1)	119 (17.3)	0.45
7	550 (79.8)	115 (16.7)	0.51
8	446 (64.6)	**	**
9	494 (71.7)	121 (17.6)	0.36
10	554 (80.3)	103 (14.9)	0.47
Average	529 (76.7)	115 (16.7)	0.45
Std. Dev.	59.9 (8.69)	N/A	N/A
COV	0.11	N/A	N/A

** Improperly machined specimen

3.4 Discussion of Results

The tensile test results are compared with the previous test results obtained with the AS4 carbon fiber reinforced 2:1 $PISO_2$/LARC-TPI, and LARC-TPI, and with literature values for AS4 carbon fiber reinforced polyetheretherketone (PEEK) and polyphenylene sulfide (PPS) composites in Figures 25 and 26. The compression test results are compared likewise in Figure 27. The test results for the $PISO_2$/LARC-TPI laminates are low and comparable to the results obtained with the 2:1 formulation evaluated previously (NASA - Advanced Thermoplastic Resins - Phase I). $PISO_2$ was selected for this work because of its superior neat resin properties and because

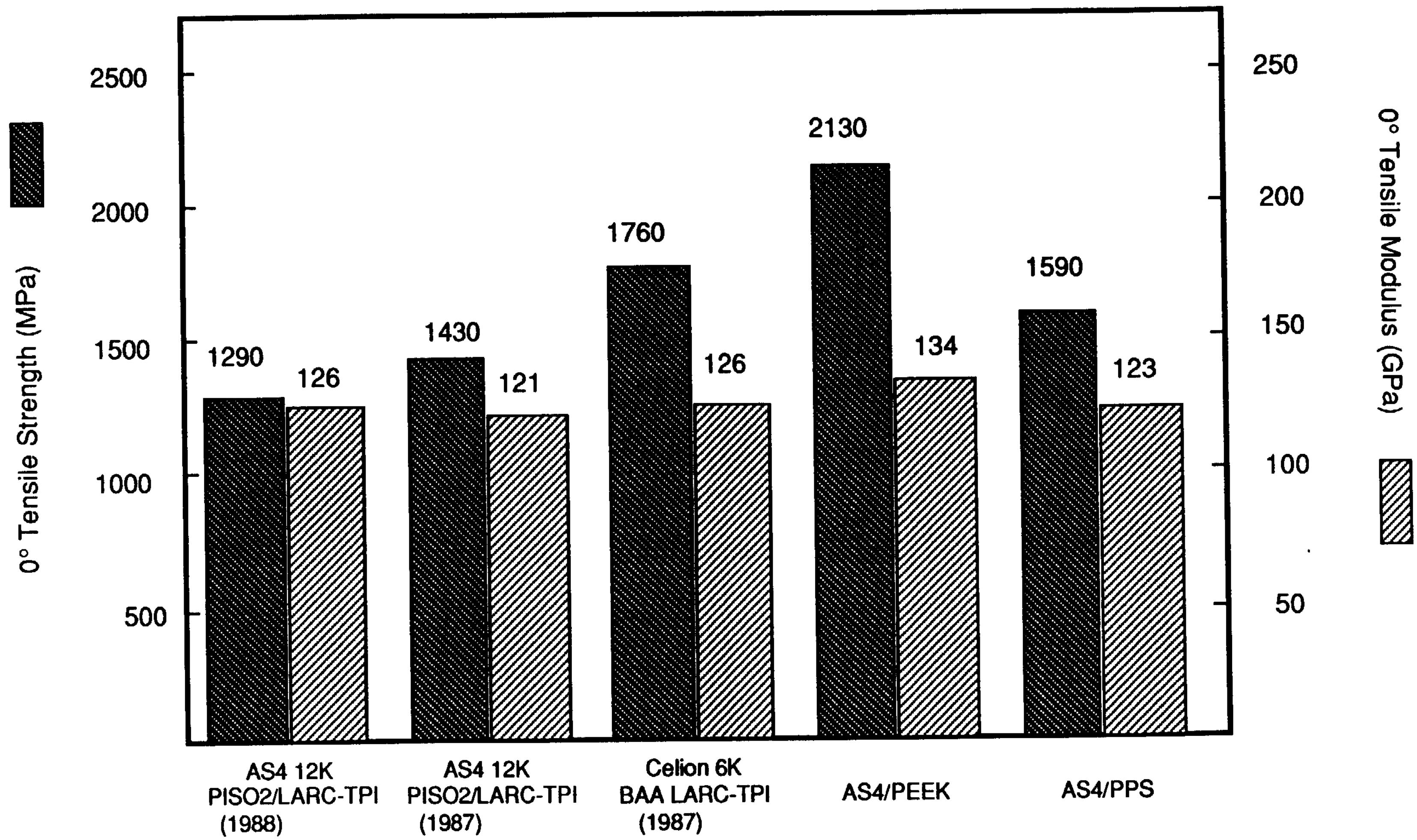


Figure 25. Comparison of Room Temperature 0 Degree Tensile Strength and Modulus

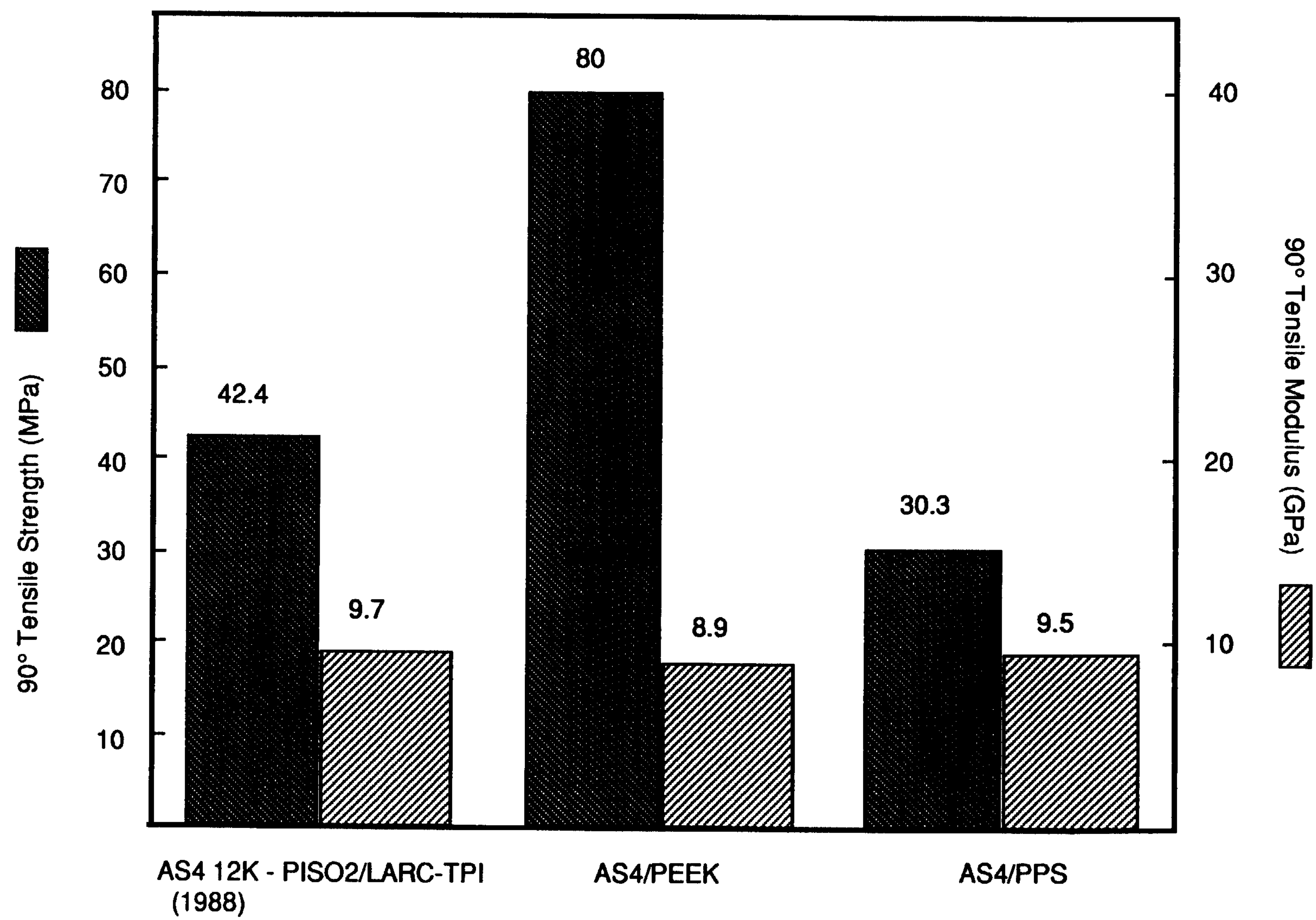


Figure 26. Comparison of Room Temperature 90 Degree Tensile Strength and Modulus

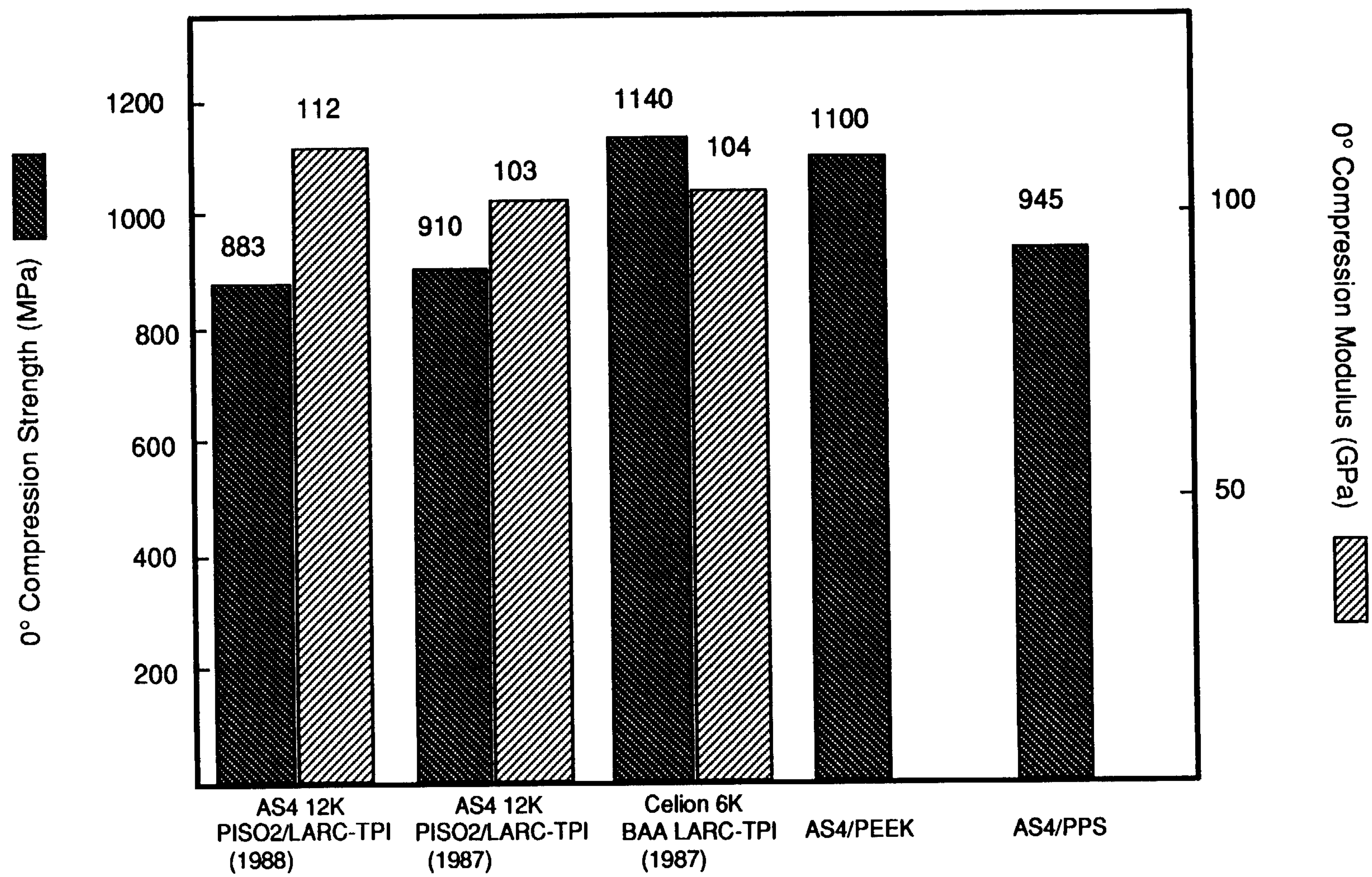
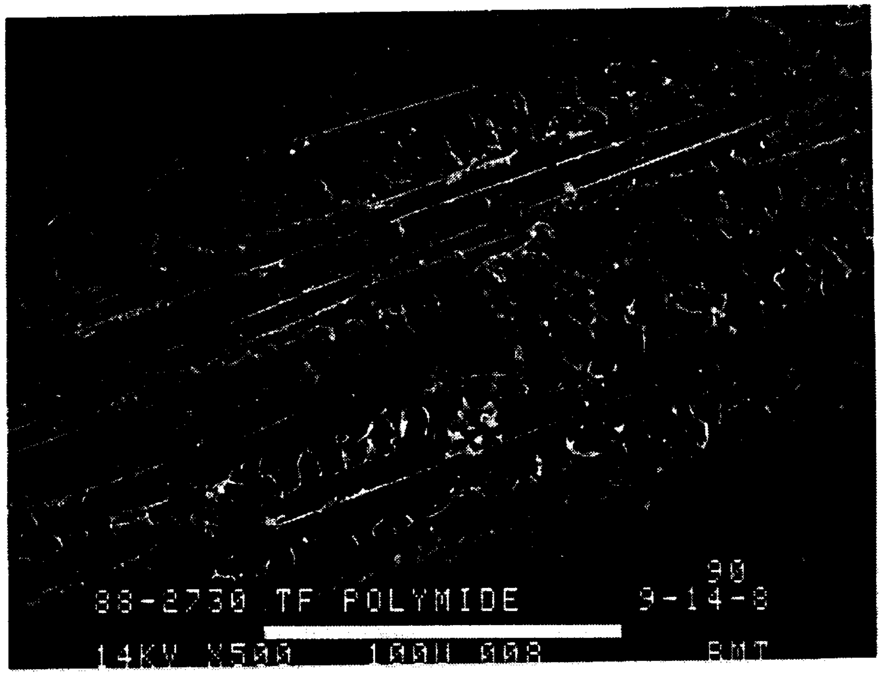


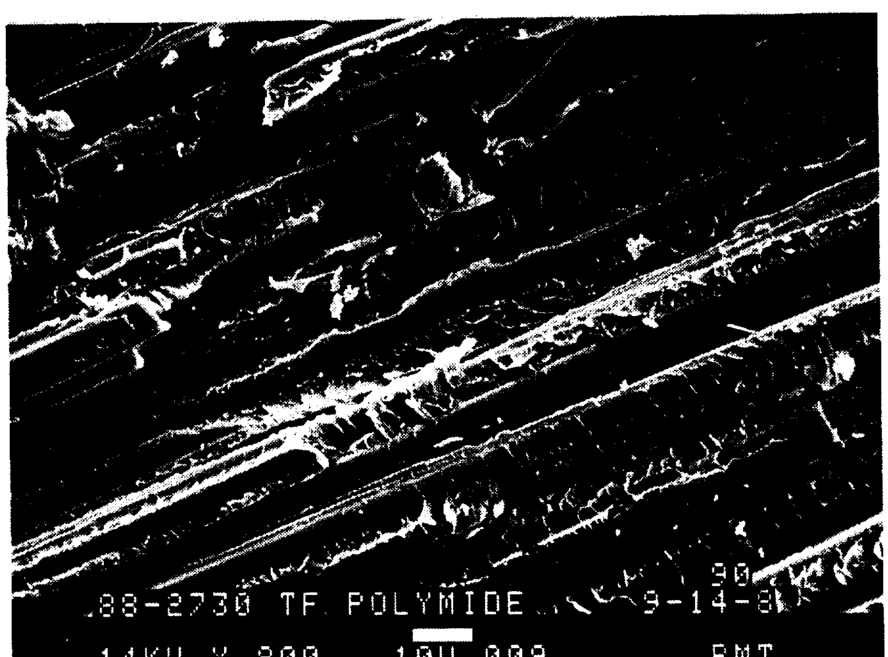
Figure 27. Comparison of Room Temperature 0 Degree Compression Strength and Modulus

preliminary testing conducted at both Boeing and NASA had indicated that $PISO_2$/LARC-TPI blends had superior composite properties.

Scanning electron micrographs (SEM) of the fracture surfaces of the 90 degree tensile test coupons (see Figures 28 and 29) show resin adhering to the unsized AS-4 carbon fibers, although there are areas with clean fibers (Figure 29(b)). Improvement of fiber/matrix adhesion would probably improve the composite mechanical properties. However, inadequate fiber/matrix adhesion does not seem to be the reason for the low mechanical properties. The processes used to produce the laminates were not optimized, and improved processing techniques might enhance composite properties.

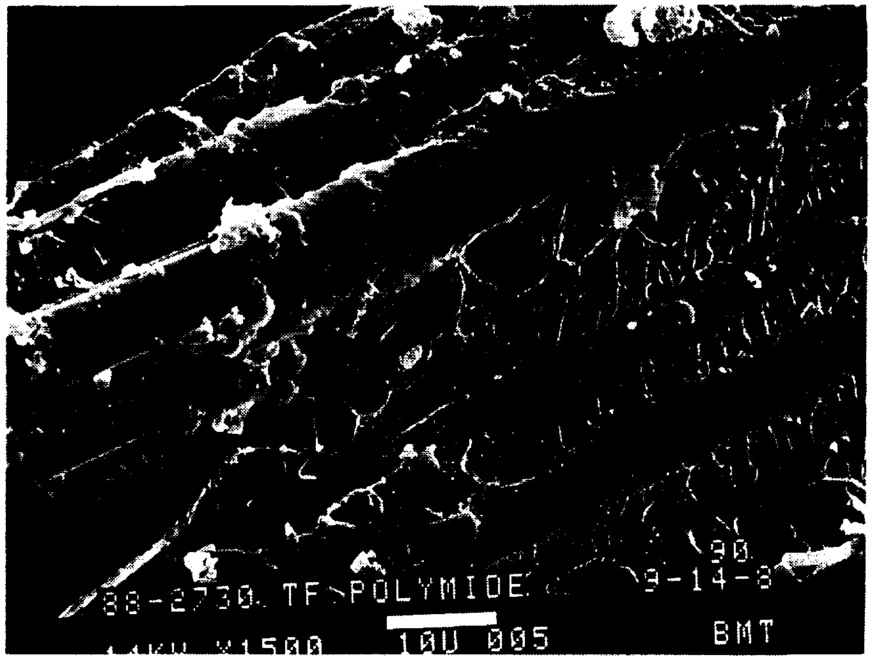


(a) 500X

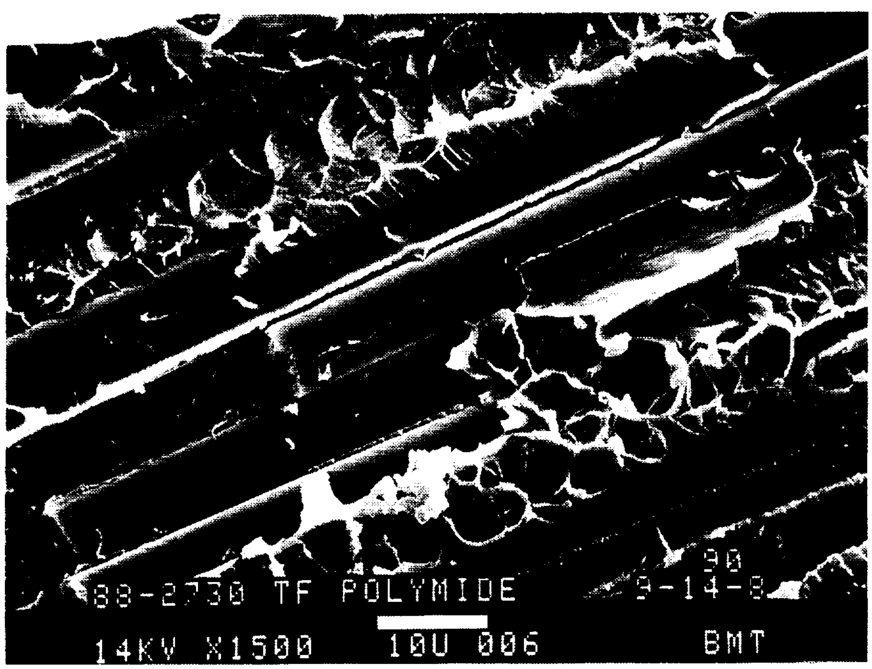


(b) 800X

Figure 28. SEM Micrographs of 0-deg Tensile Fracture Surface



(a) 1500X



(b) 1500X

Figure 29. SEM Micrographs of 0-deg Tensile Fracture Surface

4.0 Conclusions

LARC-CPI resin is an attractive high temperature adhesive for titanium bonding. The resin is very processable yielding high quality low volatile content adhesive tapes, and good lap shear strengths. Titanium lap shear strengths of up to 45.2 MPa (6550 psi) and 14.0 MPa (2030 psi) were obtained at ambient and 232°C respectively using a bonding pressure of 1.38 MPa (200 psi). Higher bond strengths at elevated temperature have been obtained by NASA. Bond strengths can be expected to decrease rapidly as the use temperature approaches the LARC-CPI glass transition temperature of 222°C.

We were unable to determine why many of our lap shear strength measurements were below test results obtained by NASA even with test specimens from the same batch. There was no apparent cause in our tape fabrication or titanium surface preparation processes, and although there were some differences between the Boeing and NASA test specimens this would not appear to account for all of the discrepancies in our results. Based on finite element analysis of the titanium lap shear test specimens used by NASA and by Boeing, the Boeing lap shear specimen could fail at a 15 to 19 percent lower apparent stress level than the NASA specimen.

There was also some variation from lot to lot of the LARC-CPI resin. We did not obtain as high lap shear strengths with later batches of resin as were obtained with the first batch. Nevertheless LARC-CPI resin is an attractive high temperature metal bonding adhesive.

LARC-TPI resin powder slurries worked well as titanium honeycomb core bonding adhesives. The keys to obtaining satisfactory bond strengths were to evenly coat the core ribbon with resin in sufficient amounts for filleting and thin enough to avoid trapping volatiles, and to use a bonding temperature that was high enough to reduce the melt viscosity. Flatwise tensile strengths of up to 4.62 MPa (670 psi) and 2.90 MPa (420 psi) were obtained at test temperatures of ambient and 232°C at a bonding pressure of 0.31 MPa (45 psi). There was a significant variation in the lot to lot processing parameters of the LARC-TPI powders. Some powders required higher bonding temperatures and pressures than others to obtain suitable bond strengths.

Although the mechanical test results obtained from poly-imidesulfone/LARC-TPI composites were comparable to results obtained earlier and were comparable to some high temperature thermoplastic composites they were not as high as desired. Average zero degree tensile and compressive strengths of 1,290 MPa (187 ksi) and 883 MPa (128 ksi) respectively were obtained at ambient temperature. LARC-TPI powders with more consistent melt flow properties are needed to repeatably obtain low void content laminates. A moderate degree of polymer adhesion to unsized AS4 carbon fibers was observed in scanning electron micrographs of fractured test coupons.

5.0 References

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Appendix A

Mechanical Test Values for Individual Test Specimens

Table A1. LARC-CPI Adhesive Bond Lap Shear Strengths as a Function of Tape Volatiles Content - Report Tables II and III

200°C tape drying temp., 2.9% volatiles, 47.3% flow. Bonding: 6.90 MPa for 15 min. @ 400°C. No postcure or heat treatment. Room Temp. Test. (Report Tables II and III)

Specimen No.	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
88-8-1	0.089 (0.004)	13.2 (1920)	100% Adhesive
88-8-2	0.127 (0.005)	27.0 (3920)	80% Adhesive
88-8-3	0.127 (0.005)	27.6 (4000)	80% Adhesive
88-8-4	0.102 (0.004)	24.2 (3510)	50% Adhesive
88-8-5	0.076 (0.003)	21.4 (3100)	100% Adhesive
Average Standard Deviation Coefficient of Variation		22.7 (3920) 5.83 (846) 0.26	80% Adhesive

200°C tape drying temp., 2.3% volatiles, 57.8% flow. Bonding: 6.90 MPa for 15 min. @ 400°C. No postcure or heat treatment. Room Temp. Test. (Report Tables II and III)

Specimen No.	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
88-8-6	0.152 (0.006)	31.2 (4530)	70% Cohesive
88-8-7	0.152 (0.006)	23.1 (3350)	50% Adhesive
88-8-8	0.178 (0.007)	23.0 (3340)	90% Adhesive
88-8-9	0.178 (0.004)	26.2 (3800)	90% Adhesive
88-8-10	0.178 (0.003)	25.1 (3640)	90% Adhesive
Average Standard Deviation Coefficient of Variation		25.7 (3730) 3.36 (487) 0.13	70% Adhesive

Table A1 (Continued). LARC-CPI Adhesive Bond Lap Shear Strengths. 250°C tape drying temp., 1.4% volatiles, 41.0% flow. Bonding: 6.90 MPa for 15 min. @ 400°C. No postcure or heat treatment. Room Temp. Test. (Report Tables II and III)

Specimen No.	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
88-8-11	0.089 (0.004)	30.1 (4360)	90% Cohesive
88-8-12	0.076 (0.003)	32.0 (4640)	90% Cohesive
88-8-13	0.064 (0.003)	33.7 (4890)	90% Cohesive
88-8-14	0.076 (0.003)	32.3 (4680)	100% Cohesive
88-8-15	0.064 (0.003)	33.0 (4780)	90% Cohesive
Average Standard Deviation Coefficient of Variation		32.2 (4670) 1.37 (199) 0.04	90% Cohesive

Table A2. LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.34% volatiles, 34.7% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. No postcure or heat treatment. Room Temp. Test. (Report Table III)

Specimen (250-1B-1)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
1	0.089 (0.0035)	41.4 (6000)	70% Cohesive
2	0.076 (0.003)	46.9 (6800)	60% Cohesive
3	0.084 (0.0033)	41.2 (5970)	70% Cohesive
4	0.071 (0.0028)	50.7 (7350)	50% Cohesive
5	0.114 (0.0045)	45.8 (6640)	50% Cohesive
	Average Std. Deviation COV	45.2 (6550) 4.01 (581) 0.09	

LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.34% volatiles, 34.7% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. Postcured @ 0.69 MPa & 300°C for 4.0 hrs. 232°C Test. (Report Table III)

Specimen (250-1B-2)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
1	0.114 (0.0045)	9.82 (1420)	90% Cohesive
2	0.127 (0.0050)	13.2 (1910)	90% Cohesive
3	0.076 (0.0030)	13.8 (2000)	90% Cohesive
4	0.102 (0.0040)	16.6 (2400)	90% Cohesive
5	0.102 (0.0040)	12.4 (1800)	50% Cohesive
	Average Std. Deviation COV	13.2 (1910) 2.43 (352) 0.18	

Table A2 (Continued). LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.12% volatiles, 11.1% flow. Bonding: 0.69 MPa for 15 min. @ 400°C. Postcured @ 0.69 MPa & 300°C for 4.0 hrs. Room Temp. Test. (Report Table III)

Specimen (250-2A-2)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
1	0.089 (0.0035)	22.6 (3270)	95% Cohesive
2	0.102 (0.0040)	18.6 (2700)	95% Cohesive
3	0.089 (0.0035)	21.5 (3120)	95% Cohesive
4	0.102 (0.0040)	18.5 (2680)	95% Cohesive
5	0.140 (0.0055)	22.2 (3220)	95% Cohesive
	Average Std. Deviation COV	20.7 (3000) 1.97 (286) 0.10	

LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.12% volatiles, 11.1% flow. Bonding: 0.69 MPa for 15 min. @ 400°C. Postcured @ 0.69 MPa & 300°C for 4.0 hrs. 177°C Test. (Report Table III)

Specimen (250-2A-1)	BondlineShear Thickness mm (in)	Strength MPa (psi)	Failure Mode
1	0.114 (0.0045)	17.9 (2600)	100% Cohesive
2	0.127 (0.0050)	14.6 (2120)	100% Cohesive
3	0.102 (0.0040)	15.9 (2300)	100% Cohesive
4	0.114 (0.0045)	13.8 (2000)	100% Cohesive
5	0.127 (0.0050)	14.1 (2040)	100% Cohesive
	Average Std. Deviation COV	15.2 (2210) 1.70 (246) 0.11	

Table A2 (Continued). LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.12% volatiles, 11.1% flow. Bonding: 0.69 MPa for 15 min. @ 400°C. Postcured @ 0.69 MPa & 300°C for 4.0 hrs. 200°C Test. (Report Table III)

Specimen (250-2A-3)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
1	0.102 (0.0040)	13.2 (1920)	100% Cohesive
2	0.089 (0.0035)	16.3 (2360)	100% Cohesive
3	0.114 (0.0045)	13.2 (1920)	100% Cohesive
4	0.114 (0.0045)	16.7 (2420)	100% Cohesive
5	0.089 (0.0035)	15.9 (2300)	100% Cohesive
	Average Std. Deviation COV	15.0 (2180) 1.69 (245) 0.11	

LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.12% volatiles, 11.1% flow. Bonding: 0.69 MPa for 15 min. @ 400°C. Postcured @ 0.69 MPa & 300°C for 4.0 hrs. 232°C Test. (Report Table III)

Specimen (250-2A-1)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
1	0.089 (0.0035)	12.7 (1840)	95% Cohesive
2	0.089 (0.0035)	12.3 (1780)	95% Cohesive
3	0.114 (0.0045)	11.3 (1640)	95% Cohesive
4	0.102 (0.0040)	9.79 (1420)	95% Cohesive
5	0.127 (0.0050)	12.4 (1800)	95% Cohesive
	Average Std. Deviation COV	11.7 (1700) 1.19 (172) 0.10	

Table A2 (Continued). LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.12% volatiles, 11.1% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. Postcured @ 0.69 MPa & 300°C for 4.0 hrs. Room Temperature Test. (Report Table III)

Specimen (250-2A-5)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
1	0.102 (0.0040)	33.9 (4920)	95% Cohesive
2	0.089 (0.0035)	35.0 (5080)	95% Cohesive
3	0.076 (0.0030)	36.3 (5270)	95% Cohesive
4	0.102 (0.0040)	32.6 (4720)	95% Cohesive
5	0.109 (0.0043)	34.1 (4920)	95% Cohesive
	Average	34.4 (4990)	
	Std. Deviation	1.41 (204)	
	COV	0.04	

LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.12% volatiles, 11.1% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. No postcure. Room Temperature Test. (Report Table III)

Specimen (250-2A-6)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
1	0.097 (0.0038)	38.6 (5600)	100% Cohesive
2	0.114 (0.0045)	39.3 (5700)	100% Cohesive
3	0.102 (0.0040)	36.1 (5240)	100% Cohesive
4	0.102 (0.0040)	37.2 (5400)	100% Cohesive
5	0.114 (0.0045)	40.6 (5880)	100% Cohesive
	Average	38.3 (5560)	
	Std. Deviation	1.73 (251)	
	COV	0.05	

Table A2 (Continued). LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.12% volatiles, 11.1% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. No postcure. Room Temp. Test. (Report Table III)

Specimen (250-2A-7)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
1	0.076 (0.0030)	35.9 (5200)	100% Cohesive
2	0.089 (0.0035)	35.7 (5180)	100% Cohesive
3	0.102 (0.0040)	29.9 (4330)	100% Cohesive
4	0.102 (0.0040)	40.7 (5900)	100% Cohesive
5	0.076 (0.0030)	42.5 (6160)	100% Cohesive
	Average Std. Deviation COV	36.9 (5350) 4.94 (716) 0.13	

LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.93% volatiles, 22.6% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. No postcure. 232°C Test. (Report Table III)

Specimen (250-2A-8)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
1	0.127 (0.0050)	4.69 (680)	100% Adhesive
2	0.102 (0.0040)	4.69 (680)	100% Adhesive
3	0.114 (0.0045)	3.52 (510)	100% Adhesive
4	0.089 (0.0035)	4.00 (580)	100% Adhesive
5	0.102 (0.0040)	4.61 (670)	100% Adhesive
	Average Std. Deviation COV	4.30 (620) 0.52 (76.1) 0.12	

Table A2 (Continued). LARC-CPI Lap Shear Strengths. 200°C tape drying temp., 0.95% volatiles, 22.4% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. No postcure. Room Temp. Test. (Report Table III)

Specimen (200-2C-1)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
1	0.102 (0.004)	46.2 (6700)	100% Cohesive
2	0.076 (0.003)	45.5 (6600)	100% Cohesive
3	0.089 (0.0035)	44.8 (6500)	100% Cohesive
4	0.089 (0.0035)	43.5 (6310)	100% Cohesive
5	0.076 (0.003)	43.4 (6300)	100% Cohesive
	Average	44.7 (6480)	
	Std. Deviation	1.21 (176)	
	COV	0.03	

LARC-CPI Lap Shear Strengths. 200°C tape drying temp., 0.93% volatiles, 22.6% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. Postcured @ 0.69 MPa & 300°C for 4.0 hrs. 232°C Test. (Report Table III)

Specimen (200-3A-1)	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
1	0.221 (0.0087)	2.76 (400)	100% Adhesive
2	0.211 (0.0083)	6.00 (870)	100% Adhesive
3	0.191 (0.0075)	2.48 (360)	100% Adhesive
4	0.196 (0.0077)	3.21 (466)	100% Adhesive
5	0.203 (0.0080)	2.83 (410)	100% Adhesive
	Average	3.46 (500)	
	Std. Deviation	1.45 (210)	
	COV	0.42	

Table A3. LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.71% volatiles, 26.6% flow. Bonding: 1.38 MPa for 10 min. @ 400°C. No postcure. Room Temp. Test. (Report Table IV)

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
8912CPI-1	0.089 (0.0035)	43.4 (6300)	100% Cohesive
8912CPI-2	0.102 (0.0040)	40.0 (5800)	100% Cohesive
8912CPI-3	0.102 (0.0040)	35.2 (5100)	100% Cohesive
8912CPI-4	0.132 (0.0052)	37.6 (5450)	100% Cohesive
8912CPI-5	0.127 (0.0050)	40.5 (5870)	100% Cohesive
	Average	39.3 (5700)	
	Std. Deviation	3.12 (453)	
	COV	0.08	

LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.34% volatiles, 34.7% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. Postcured at 0.69 MPa and 300°C for 4.0 hours. Heat treatment at 316°C for 100 hours. Room Temp. Test. (Report Table IV)

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
897CPI-6	0.142 (0.0056)	16.6 (2400)	95% Adhesive
897CPI-7	0.142 (0.0056)	15.4 (2240)	95% Adhesive
897CPI-8	0.142 (0.0056)	13.9 (2020)	95% Adhesive
897CPI-9	0.142 (0.0056)	17.1 (2480)	95% Adhesive
897CPI-10	0.142 (0.0056)	17.9 (2600)	95% Adhesive
	Average	16.2 (2350)	
	Std. Deviation	1.55 (225)	
	COV	0.10	

Table A3 (Continued). LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.34% volatiles, 34.7% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. Postcured at 0.69 MPa and 300°C for 4.0 hours. Heat treatment at 316°C for 100 hours. 232°C Test. (Report Table IV)

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
897CPI-11	0.145 (0.0057)	14.8 (2140)	60% Cohesive
897CPI-12	0.142 (0.0056)	15.3 (2220)	50% Cohesive
897CPI-13	0.145 (0.0057)	14.9 (2160)	40% Cohesive
897CPI-14	0.145 (0.0057)	12.8 (1850)	60% Cohesive
897CPI-15	0.145 (0.0057)	12.1 (1760)	70% Cohesive
	Average	14.0 (2030)	
	Std. Deviation	1.42 (206)	
	COV	0.10	

LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.12% volatiles, 11.1% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. Postcured at 0.69 MPa and 300°C for 4.0 hours. Heat treatment at 316°C for 500 hours. Room Temp. Test. (Report Table IV)

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
89CPI-11	0.107 (0.0042)	6.28 (910)	95% Adhesive
89CPI-12	0.076 (0.0030)	3.93 (570)	95% Adhesive
89CPI-13	0.089 (0.0035)	5.52 (800)	95% Adhesive
89CPI-14	0.089 (0.0035)	5.42 (786)	95% Adhesive
89CPI-15	0.076 (0.0030)	5.93 (860)	95% Adhesive
	Average	5.41 (785)	
	Std. Deviation	0.897 (130)	
	COV	0.17	

Table A3 (Continued). LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.12% volatiles, 11.1% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. Postcured at 0.69 MPa and 300°C for 4.0 hours. Heat treatment at 316°C for 500 hours. 232°C Test. (Report Table IV)

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
89CPI-16	0.140 (0.0055)	4.41 (640)	95% Adhesive
89CPI-17	0.102 (0.0040)	3.31 (480)	95% Adhesive
89CPI-18	0.127 (0.0050)	4.61 (668)	95% Adhesive
89CPI-19	0.114 (0.0045)	4.58 (664)	95% Adhesive
89CPI-20	0.089 (0.0035)	4.66 (676)	95% Adhesive
	Average	4.32 (626)	
	Std. Deviation	0.569 (82.5)	
	COV	0.13	

LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.51% volatiles, 26.1% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. Postcured at 0.69 MPa and 300°C for 4.0 hours. Heat treatment at 316°C for 100 hours. Room Temp. Test. (Report Table IV)

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
89IRD-21	0.127 (0.0050)	13.8 (2000)	100% Adhesive
89IRD-22	0.127 (0.0050)	13.8 (2000)	100% Adhesive
89IRD-23	0.127 (0.0050)	12.4 (1800)	100% Adhesive
89IRD-24	0.127 (0.0050)	11.9 (1720)	100% Adhesive
89IRD-25	0.140 (0.0055)	12.1 (1760)	100% Adhesive
	Average	12.8 (1860)	
	Std. Deviation	0.93 (134)	
	COV	0.07	

Table A3 (Continued). LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.51% volatiles, 26.1% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. Postcured at 0.69 MPa and 300°C for 4.0 hours. Heat treatment at 316°C for 100 hours. 200°C Test. (Report Table IV)

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
89IRD-31	0.119 (0.0047)	14.1 (2040)	50-70% Cohesive
89IRD-32	0.127 (0.0050)	14.6 (2120)	50-70% Cohesive
89IRD-33	0.140 (0.0055)	13.5 (1960)	50-70% Cohesive
89IRD-34	0.127 (0.0050)	13.3 (1930)	50-70% Cohesive
89IRD-35	0.114 (0.0045)	13.0 (1880)	50-70% Cohesive
	Average Std. Deviation COV	13.7 (1990) 0.65 (94.8) 0.05	

LARC-CPI Lap Shear Strengths. 250°C tape drying temp., 0.51% volatiles, 26.1% flow. Bonding: 1.38 MPa for 15 min. @ 400°C. Postcured at 0.69 MPa and 300°C for 4.0 hours. Heat treatment at 316°C for 100 hours. 232°C Test. (Report Table IV)

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
89IRD-26	0.140 (0.0055)	14.6 (2120)	50-60% Cohesive
89IRD-27	0.152 (0.0060)	11.9 (1720)	50-60% Cohesive
89IRD-28	0.102 (0.0040)	13.6 (1970)	50-60% Cohesive
89IRD-29	0.152 (0.0060)	14.2 (2060)	50-60% Cohesive
89IRD-30	0.127 (0.0050)	13.8 (2000)	50-60% Cohesive
	Average Std. Deviation COV	13.6 (1970) 1.06 (153) 0.08	

Table A4. Ti Lap Shear Strengths of Specimens Bonded and Tested at NASA-Langley. 250°C tape drying temp., 0.51% volatiles, 26.1% flow. Bonding: 2.76 MPa for 15 min. at 375°C. (Report Table V)

Specimen	Bond line (mils)	Test Temp. (°C)	Heat Treatment	Lap Shear Strength MPa (psi)	Failure* Mode
NASA-1	5.6	25	None	30.6 (4440)	90% C
NASA-6	9.7	25	None	18.7 (2710)	90% A
NASA-2	5.4	200	None	22.8 (3305)	60% C
NASA-9	8.9	200	None	21.0 (3045)	60% C
NASA-8	9.7	200	100 hrs @ 316°C	21.0 (3051)	90% C
NASA-11	7.9	200	100 hrs @ 316°C	23.5 (3401)	90% C
NASA-3	6.1	232	None	7.76 (1125)	100% A
NASA-12	7.7	232	None	10.5 (1525)	100% A
NASA-4	6.8	232	18 hrs @ 316°C	17.3 (2510)	60% C
NASA-10	8.0	232	18 hrs @ 316°C	14.8 (2152)	60% C
NASA-5	10.6	232	100 hrs @ 316°C	18.6 (2700)	100% C
NASA-7	10.7	232	100 hrs @ 316°C	17.2 (2499)	70% C

*C = Cohesive Fracture, A = Adhesive Fracture.

Table A5. Ti Lap Shear Strengths of Specimens Bonded and Tested at Boeing. 250°C tape drying temp., 0.51% volatiles, 26.1% flow. Bonding: 1.38 MPa for 15 min. at 375°C. (Report Table VI)

Specimen	Bond line (mils)	Test Temp. (°C)	Heat Treatment	Lap Shear Strength MPa (psi)	Failure Mode
589-1	8.0	25	None	3.72 (540)	100% A
589-2	7.0	25	None	8.02 (1164)	100% A
589-3	8.5	25	None	6.39 (926)	100% A
589-4	9.0	232	None	7.45 (1080)	100% A
589-5	8.0	232	None	7.17 (1040)	100% A
589-6	7.0	232	None	6.34 (920)	100% A
589-7	7.0	232	100 hrs @ 316°C	12.5 (1810)	90% C
589-8	Specimen came apart before testing.				
589-9	9.0	232	100 hrs @ 316°C	11.2 (1620)	90% C
589-10	7.5	232	100 hrs @ 316°C	13.1 (1900)	90% C

*C = Cohesive Fracture, A = Adhesive Fracture.

Table A6. Lap Shear Strengths of Titanium Specimens Bonded with LARC-CPI at NASA and Tested at Boeing. Bonding: 1.38 MPa for 30 min. at 375°C. LARC-CPI resin solution batch number SH 69-67-1. Tape drying temperature: 200°C; volatiles content: 2.8%. (Report Table VII)

16 Hours @ 308°C. No Heat Treatment at 316°C; Tested at Ambient.

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
N-557	0.267 (0.0105)	13.9 (2010)	70% Cohesive
N-558	0.254 (0.0100)	13.3 (1930)	60% Cohesive
N-559	0.241 (0.0095)	16.0 (2320)	70% Cohesive
N-560	0.221 (0.0087)	16.0 (2320)	50% Cohesive
N-561	0.183 (0.0072)	18.8 (2720)	50% Cohesive
	Average	15.6 (2260)	
	Std. Deviation	2.15 (312)	
	COV	0.14	

LARC-CPI Lap Shear Strengths. 16 Hours @ 308°C. No Heat Treatment at 316°C; Tested at 200°C (392°F).

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
N-562	0.191 (0.0075)	14.6 (2120)	80% Adhesive
N-563	0.165 (0.0065)	11.7 (1700)	80% Adhesive
N-564	0.152 (0.0060)	13.0 (1884)	40% Adhesive
N-565	0.152 (0.0060)	13.0 (1880)	40% Adhesive
N-566	0.119 (0.0047)	12.1 (1750)	80% Cohesive
	Average	12.9 (1870)	
	Std. Deviation	1.12 (163)	
	COV	0.09	

Table A6 (Continued). LARC-CPI Lap Shear Strengths. 16 Hours @ 308°C. Heat Treatment at 316°C for 100 Hours; Tested at Ambient.

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
N-567	0.165 (0.0065)	22.1 (3200)	60% Cohesive
N-568	0.178 (0.0070)	17.7 (2560)	80% Adhesive
N-569	0.191 (0.0075)	17.1 (2480)	80% Adhesive
N-570	0.165 (0.0065)	21.9 (3170)	80% Adhesive
N-571	0.152 (0.0060)	20.1 (2920)	80% Adhesive
	Average Std. Deviation COV	19.8 (2870) 1.12 (335) 0.12	

Table A6 (Continued). LARC-CPI Lap Shear Strengths. 16 Hours @ 308°C. Heat Treatment at 316°C for 100 Hours; Tested at 200°C (392°F).

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
N-572	0.127 (0.0050)	25.4 (3680)	80% Cohesive
N-573	0.165 (0.0065)	25.4 (3680)	80% Cohesive
N-574	0.178 (0.0070)	20.7 (3000)	50% Cohesive
N-575	0.216 (0.0085)	20.4 (2960)	50% Cohesive
N-576	0.152 (0.0060)	19.3 (2800)	80% Adhesive
	Average Std. Deviation COV	22.2 (3220) 2.92 (423) 0.13	

Table A6 (Continued). LARC-CPI Lap Shear Strengths. 16 Hours @ 308°C. Heat Treatment at 316°C for 100 Hours; Tested at 32°C (450°F).

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
N-577	0.228 (0.0090)	11.3 (1640)	50% Adhesive
N-578	0.203 (0.0080)	10.6 (1530)	80% Adhesive
N-579	0.191 (0.0075)	12.7 (1840)	80% Adhesive
N-581	0.165 (0.0065)	10.8 (1560)	80% Adhesive
N-582	0.152 (0.0060)	11.0 (1600)	80% Adhesive
	Average	11.2 (1630)	
	Std. Deviation	0.841 (122)	
	COV	0.07	

Table A7. LARC-TPI (Rogers TPI powder) Titanium Lap Shear Strengths (Report Section 2.2.3)

Specimen	Bondline Thickness mm (in)	Shear Strength MPa (psi)	Failure Mode
12.4 MPa Bonding Pressure:			
1	0.152 (0.006)	29.0 (4200)	100% Cohesive
2	0.127 (0.005)	29.7 (4300)	100% Cohesive
3	0.127 (0.005)	27.3 (3960)	100% Cohesive
4	0.152 (0.006)	28.6 (4150)	90% Cohesive
5	0.127 (0.005)	25.2 (3660)	90% Cohesive
Average		28.0 (4054)	100% Cohesive
Std. Dev.		1.74 (253)	
COV		0.06	
6.90 MPa Bonding Pressure:			
1	0.152 (0.006)	30.3 (4400)	100% Cohesive
2	0.140 (0.006)	21.0 (3050)	100% Cohesive
3	0.140 (0.006)	24.6 (3560)	100% Cohesive
4	0.140 (0.006)	26.9 (3900)	100% Cohesive
5	0.191 (0.008)	29.7 (4300)	95% Cohesive
Average		26.5 (3840)	100% Cohesive
Std. Dev.		3.83 (555)	
COV		0.14	

Table A8. LARC-CPI Titanium Lap Shear Strengths - Boeing Process Development

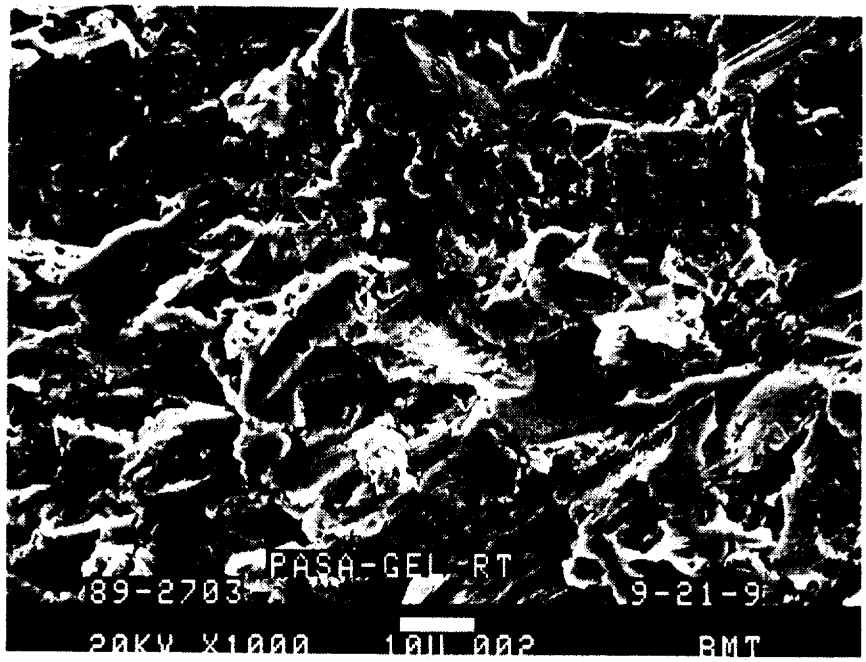
Drying Temp. (°C)	Vol. Flow		Bond Press MPa	Test Temp. (°C)	Average Strength MPa (psi)	Failure Surface
	(wt%)	(wt%)				
250	0.51	26.1	1.38	ambient	12.8(1860)	100% Adh
250	0.51	26.1	1.38	200°C	13.7(1990)	50-70% Coh
250	0.51	26.1	1.38	232°C	13.6(1970)	50-60% Coh

Notes:

1. LARC-CPI resin solution batch number SH 39-92-17.
2. Postured for 4 hours at 300°C and 0.69 MPa, followed by heat treatment for 100 hours at 316°C and atmospheric pressure.
3. Air flow rate in the heat treatment oven was 4.13 m^3/min (146 ft^3/min).

Appendix B

Scanning Electron Micrographs - Titanium



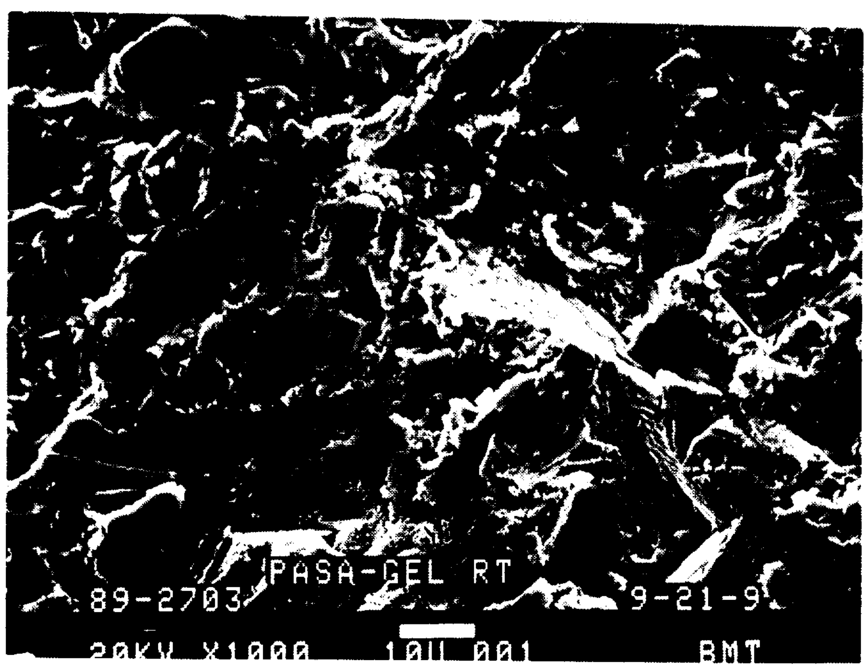
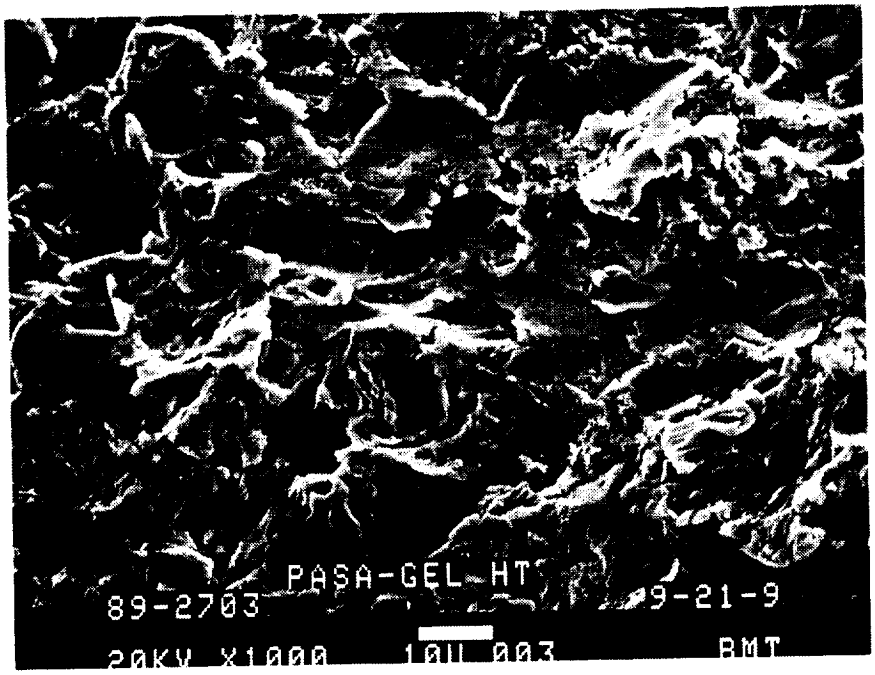


Figure B1. SEM Micrographs of Titanium Foil Treated With Pasa-Jell 107 (1,000X)



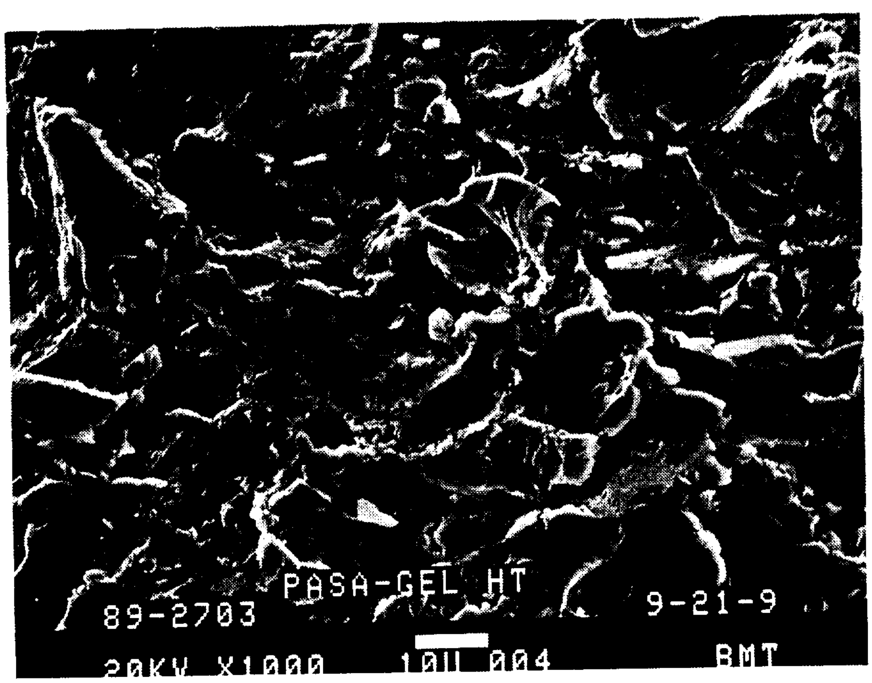
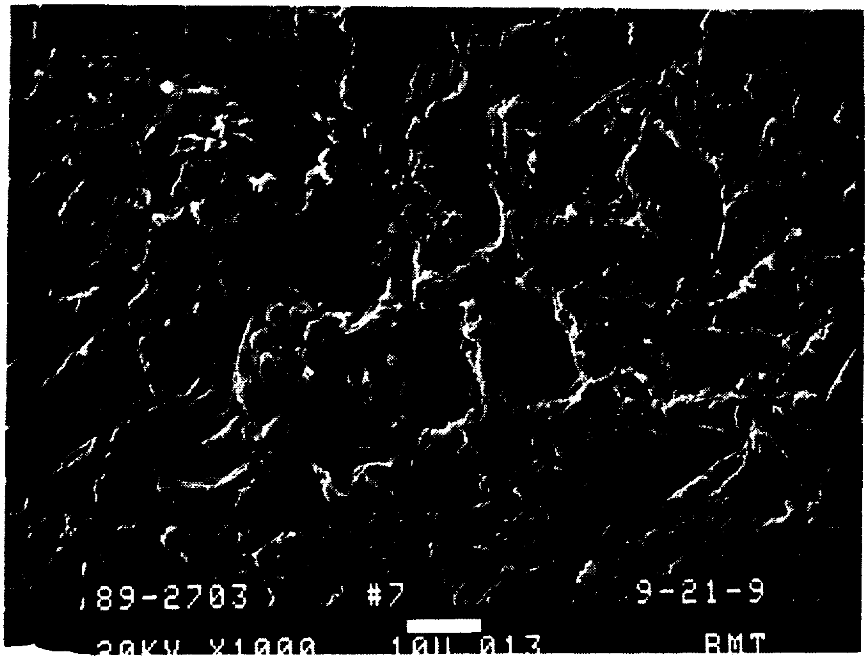


Figure B2. SEM Micrographs of Titanium Foil Treated With Pasa-Jell 107 and Heated to 400°C (750°F) for 15 min (1,000X)



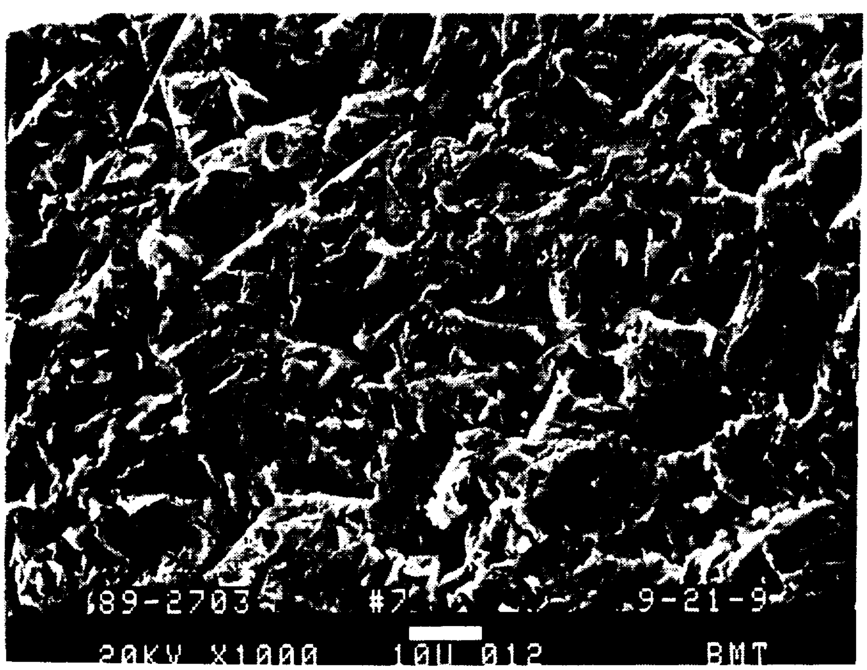
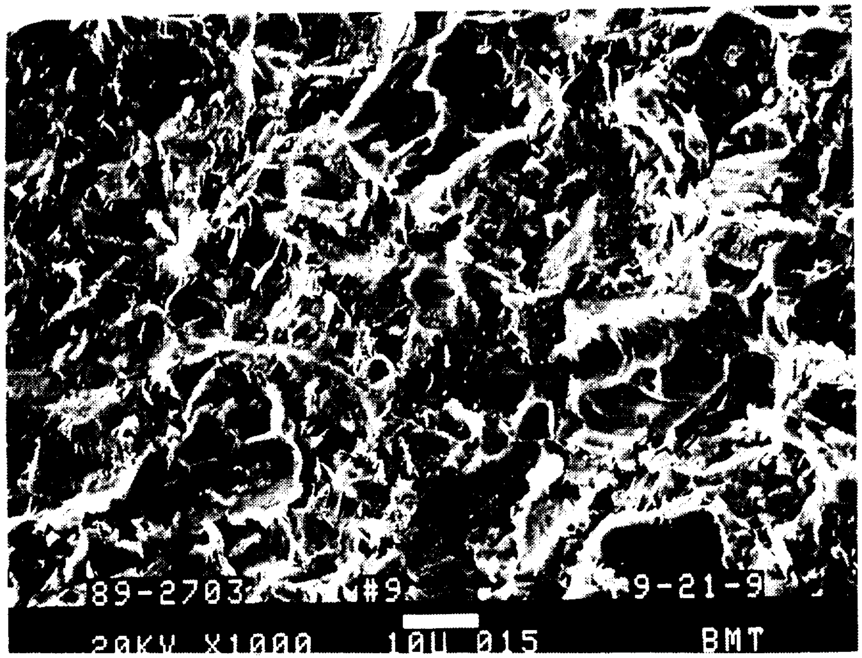


Figure B3. SEM Micrographs of Adhesive Failure Surface of Titanium Lap Shear Specimen No. NASA-7 (1,000X)



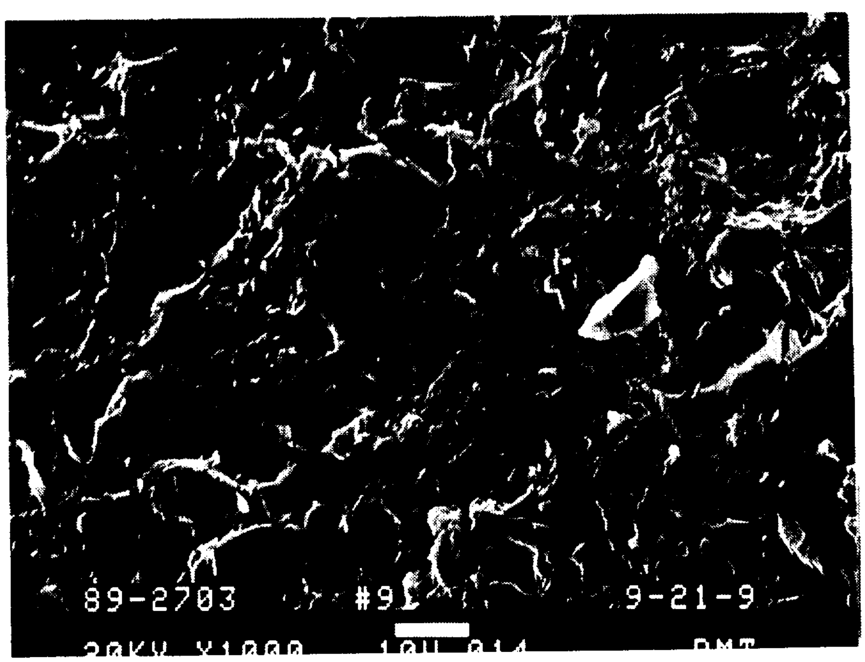
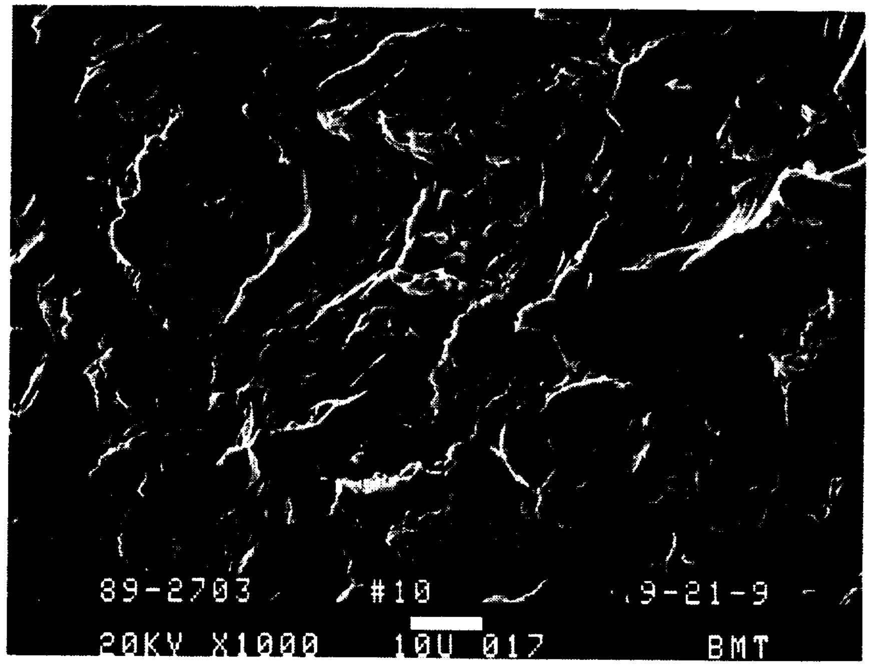


Figure B4. SEM Micrographs of Adhesive Failure Surface of Titanium Lap Shear Specimen No. 897CPI-9 (1,000X)



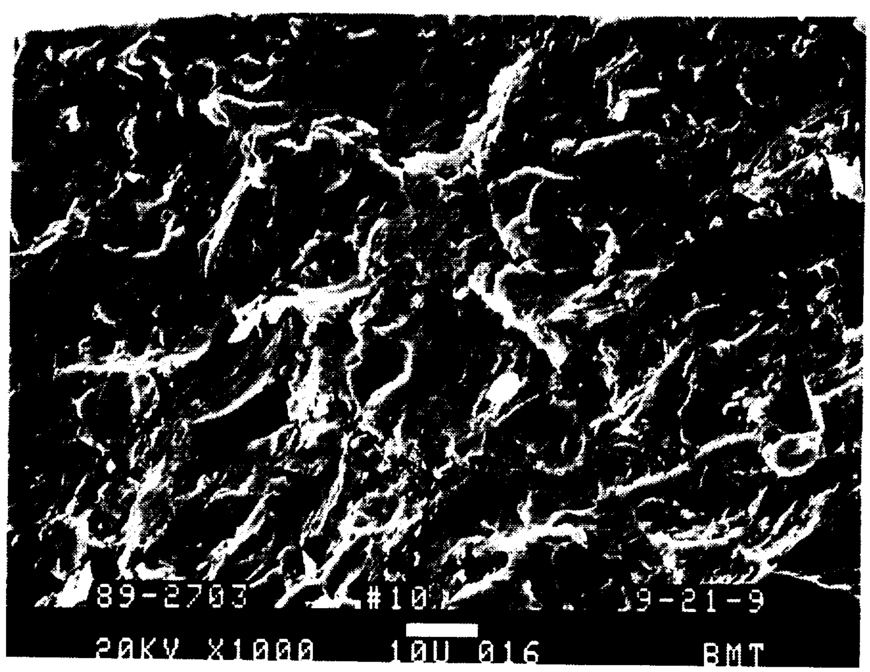
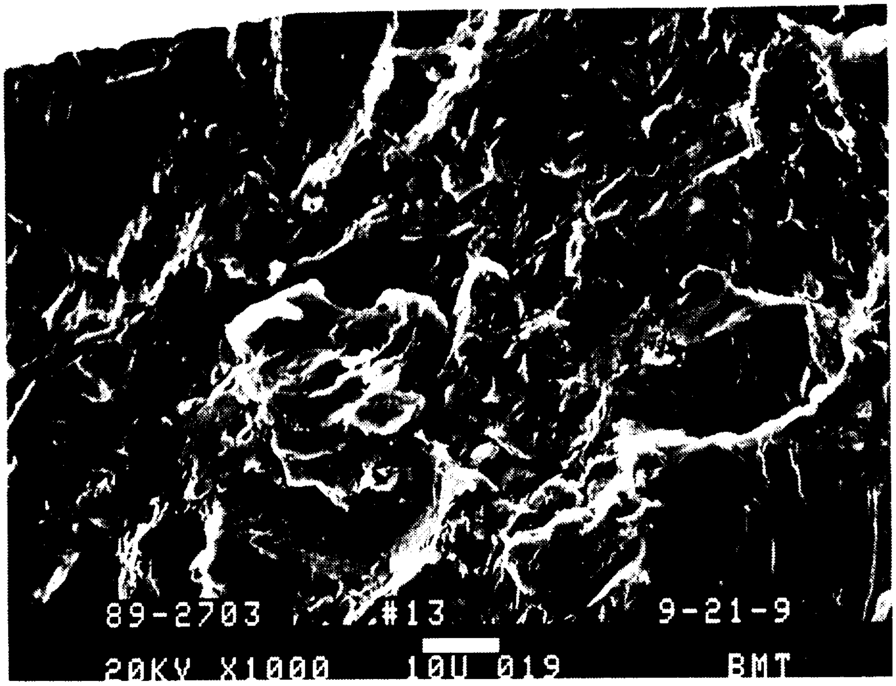


Figure B5. SEM Micrographs of Adhesive Failure Surface of Titanium Lap Shear Specimen No. 589-10 (1,000X)



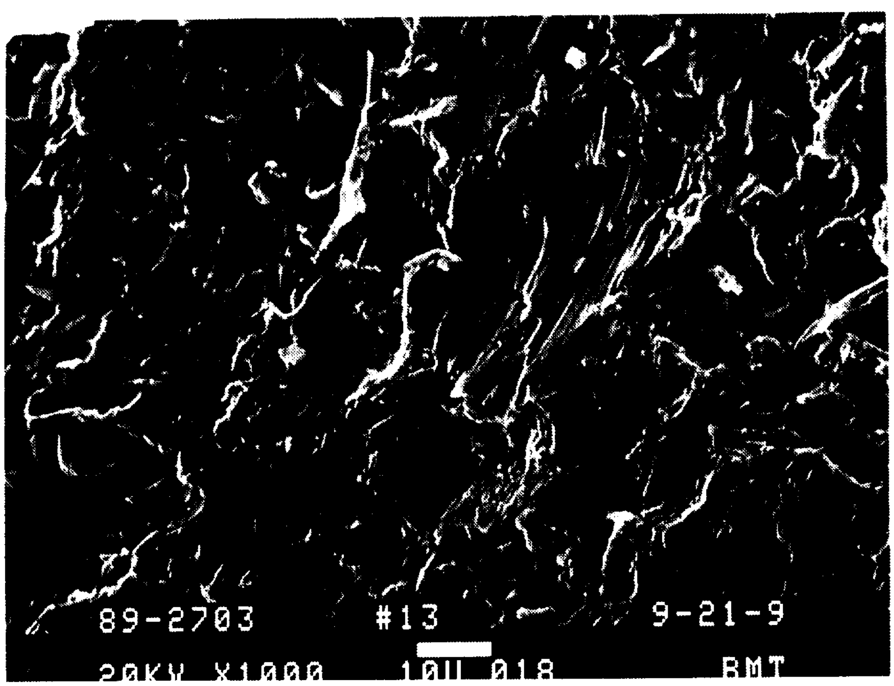
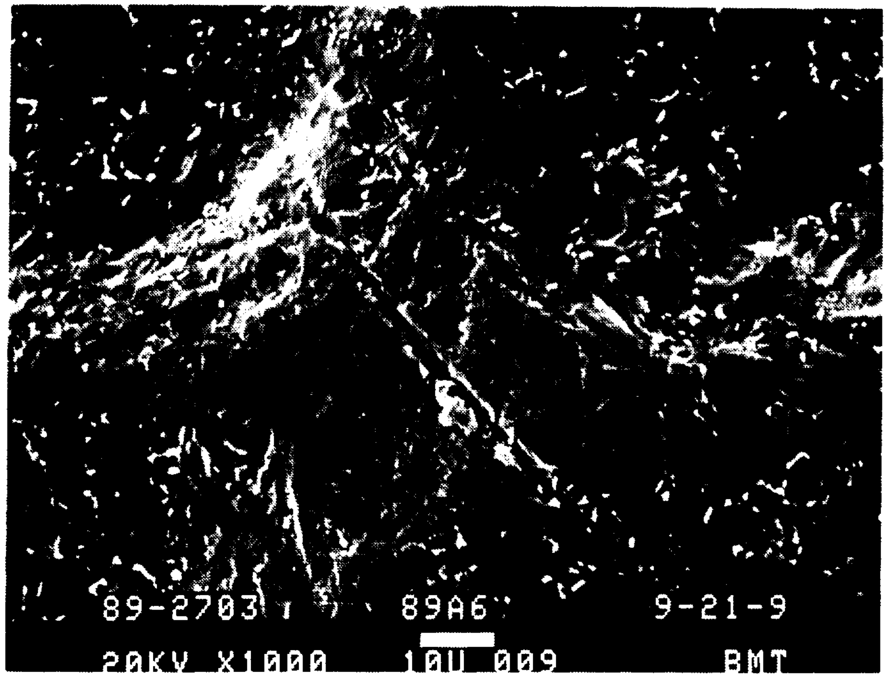


Figure B6. SEM Micrographs of Adhesive Failure Surface of Titanium Lap Shear Specimen No. 897CPI-13 (1,000X)



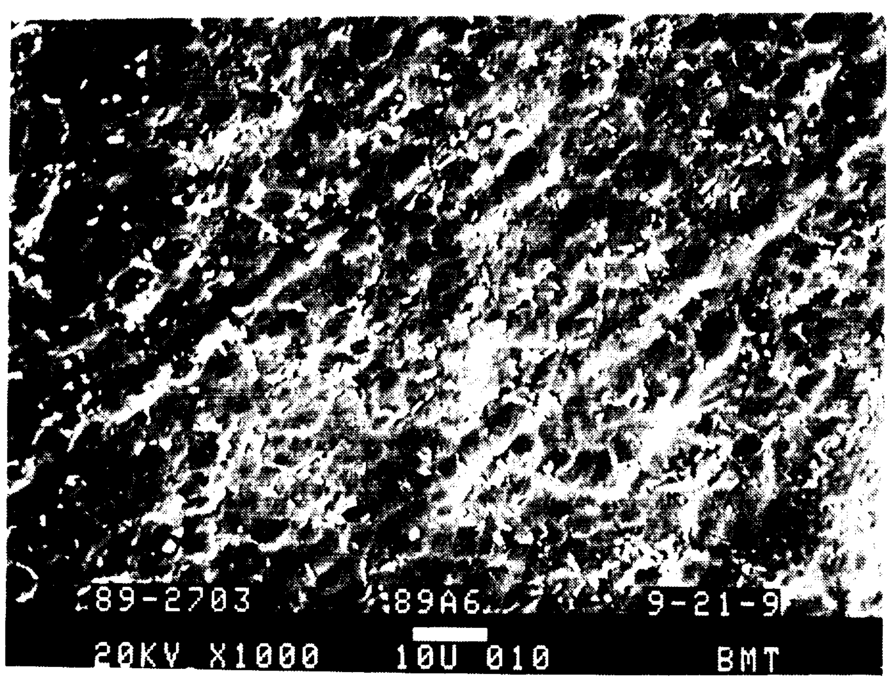


Figure B7. SEM Micrographs of Titanium Lap Shear Panel Treated With Nitric/Hydrofluoric Acid Pickle for 90 sec (1,000X)

Appendix C

Through Transmission Ultrasonic Scans of Composite Laminates

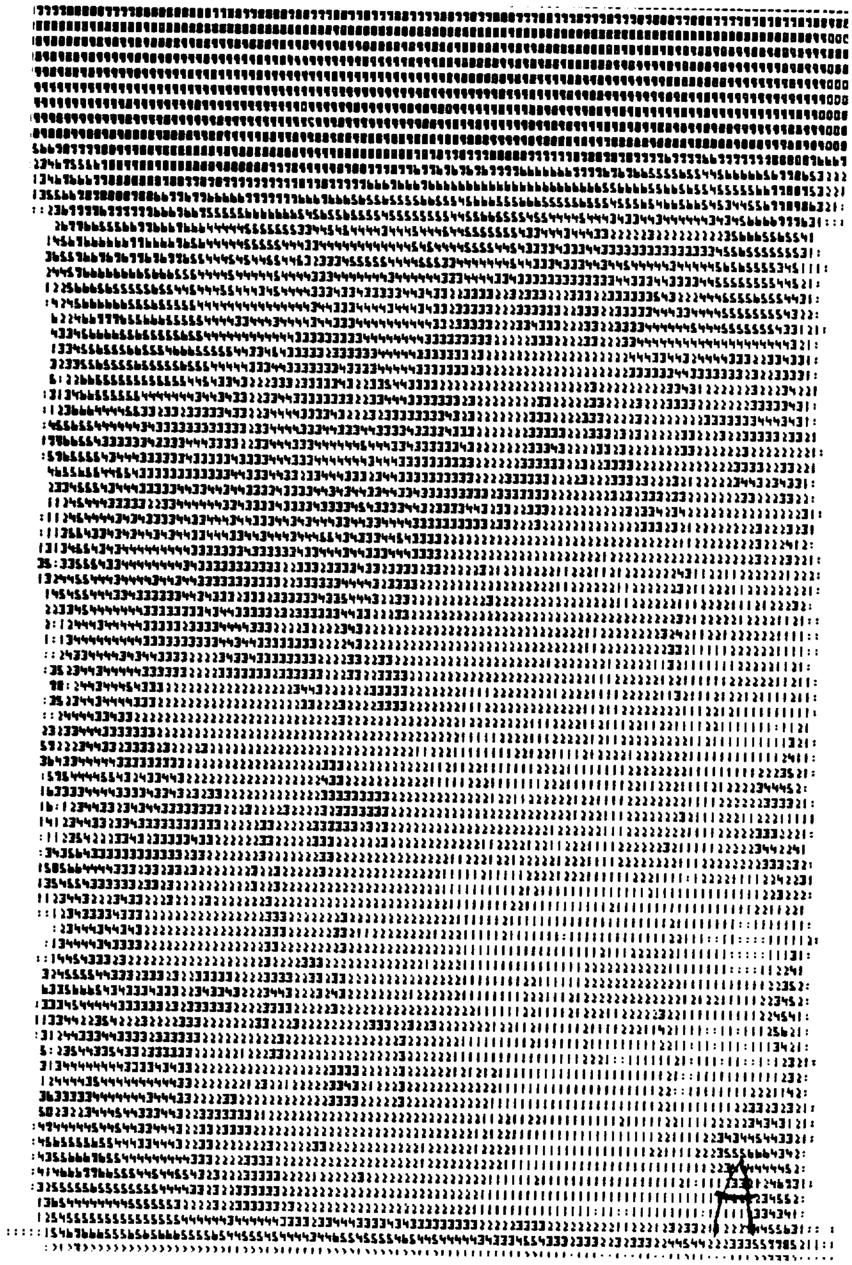


Figure C1. TTU Scan of Laminate 886A1-DT623-02, (0/90)4s

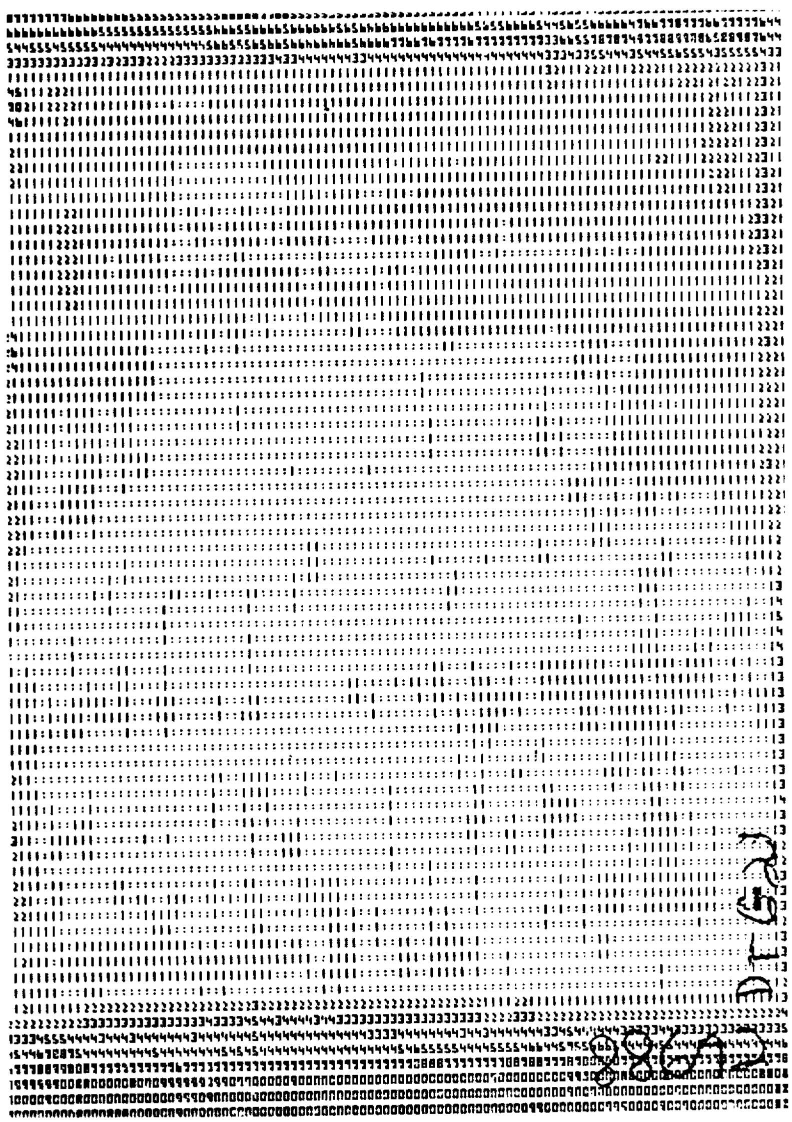


Figure C2. TTU Scan of Laminate 886A2-DT623-02, (0/90)4s

Figure C3. TTU Scan of Laminate 886A3-DT623-02, (0/90)8s

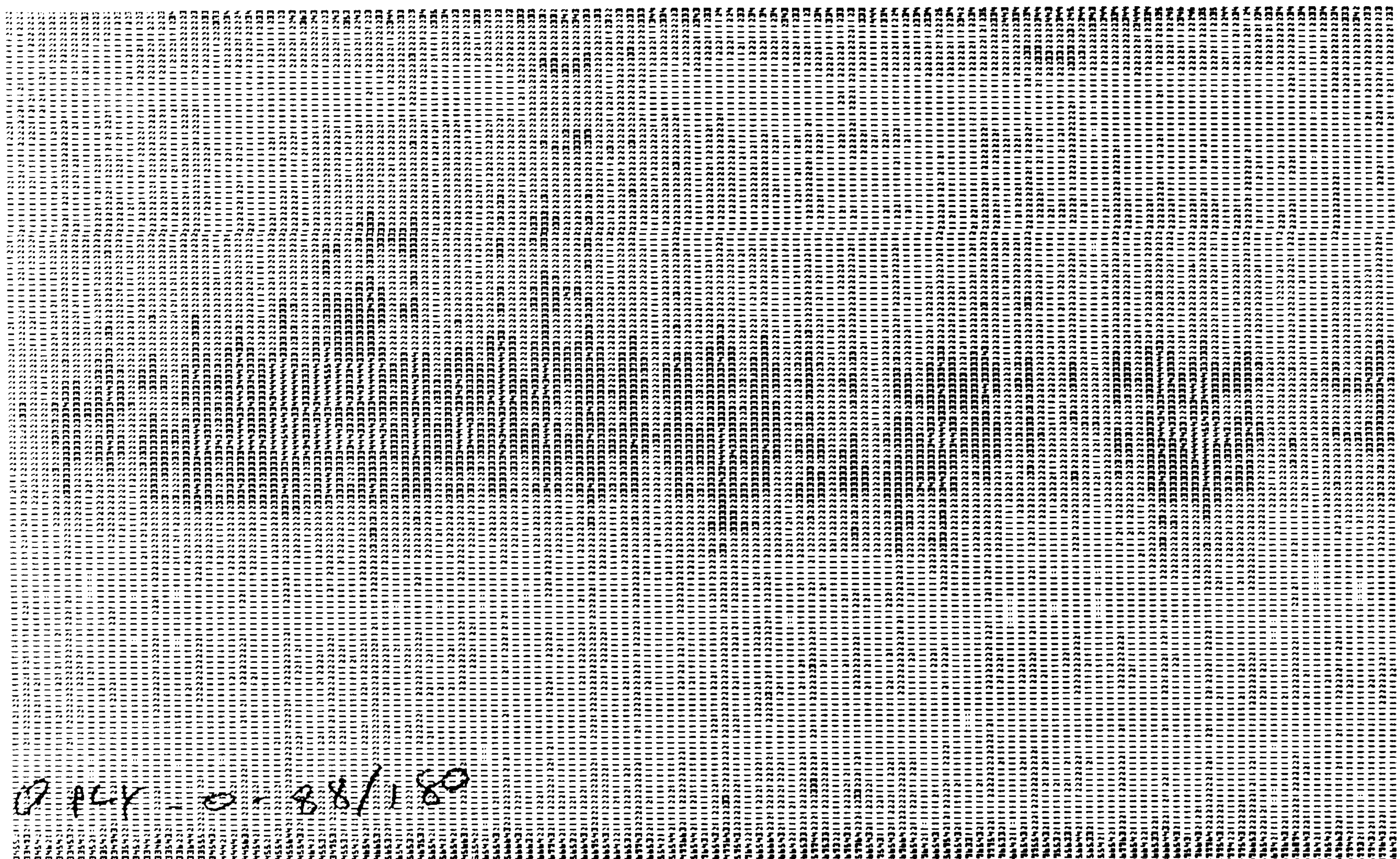


Figure C4. TTU Scan of Laminate 886B1-DT623-02, (0)10

Figure C5. TTU Scan of Laminate 886B2-DT623-02, (0)8

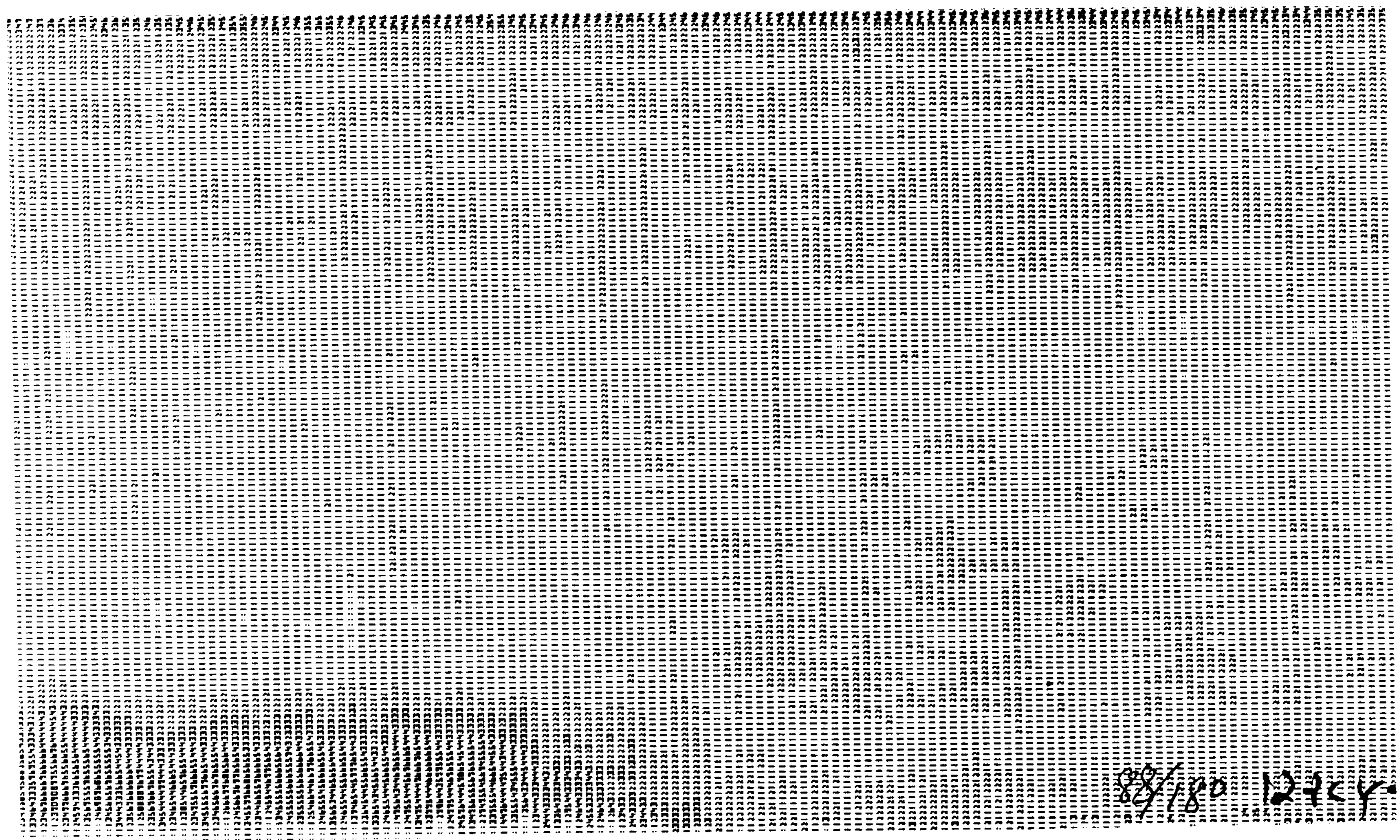


Figure C6. TTU Scan of Laminate 886B3-DT623-02, (0)12

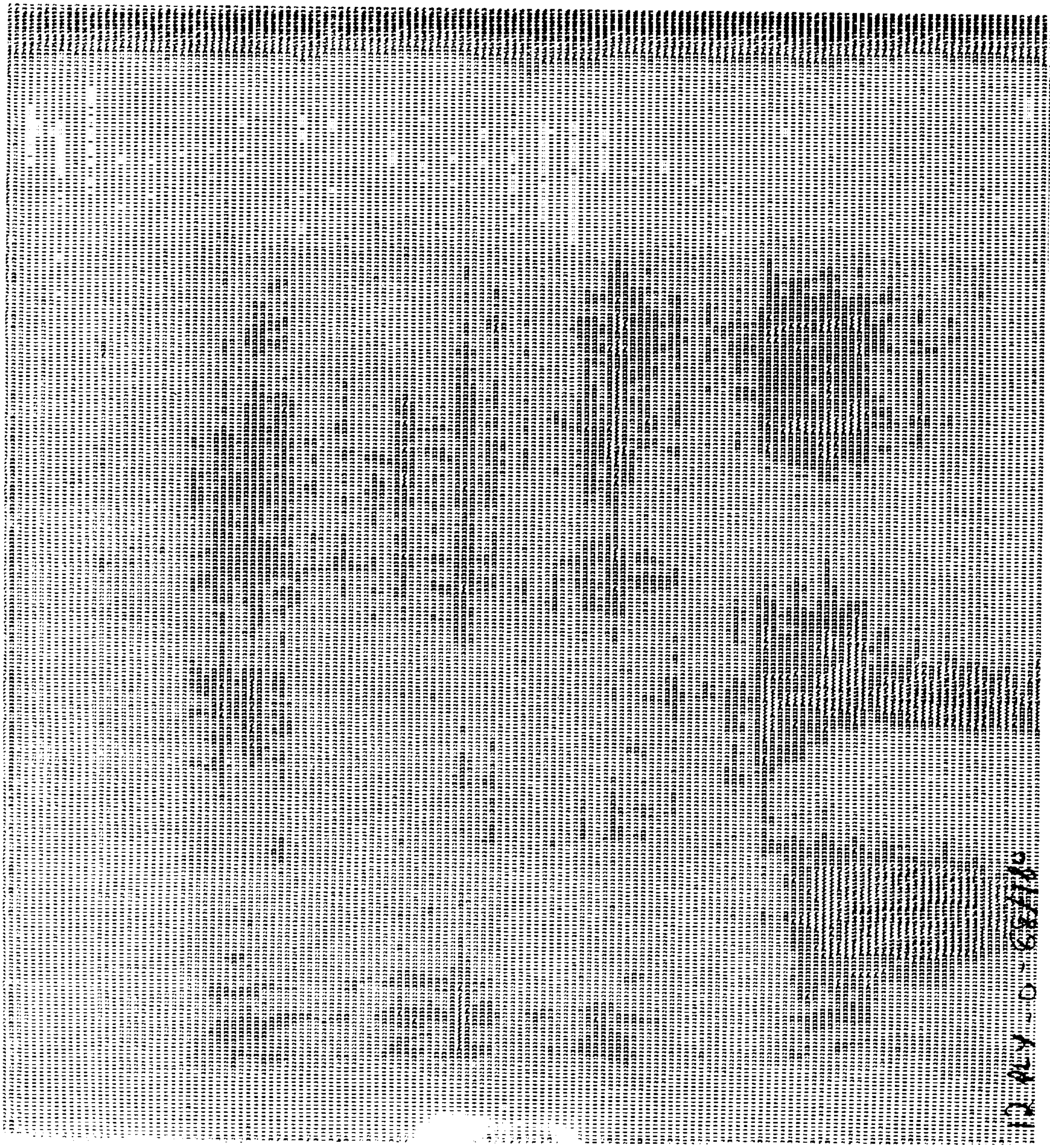


Figure C7. TTU Scan of Laminate 886B4-DT623-02, (0)12

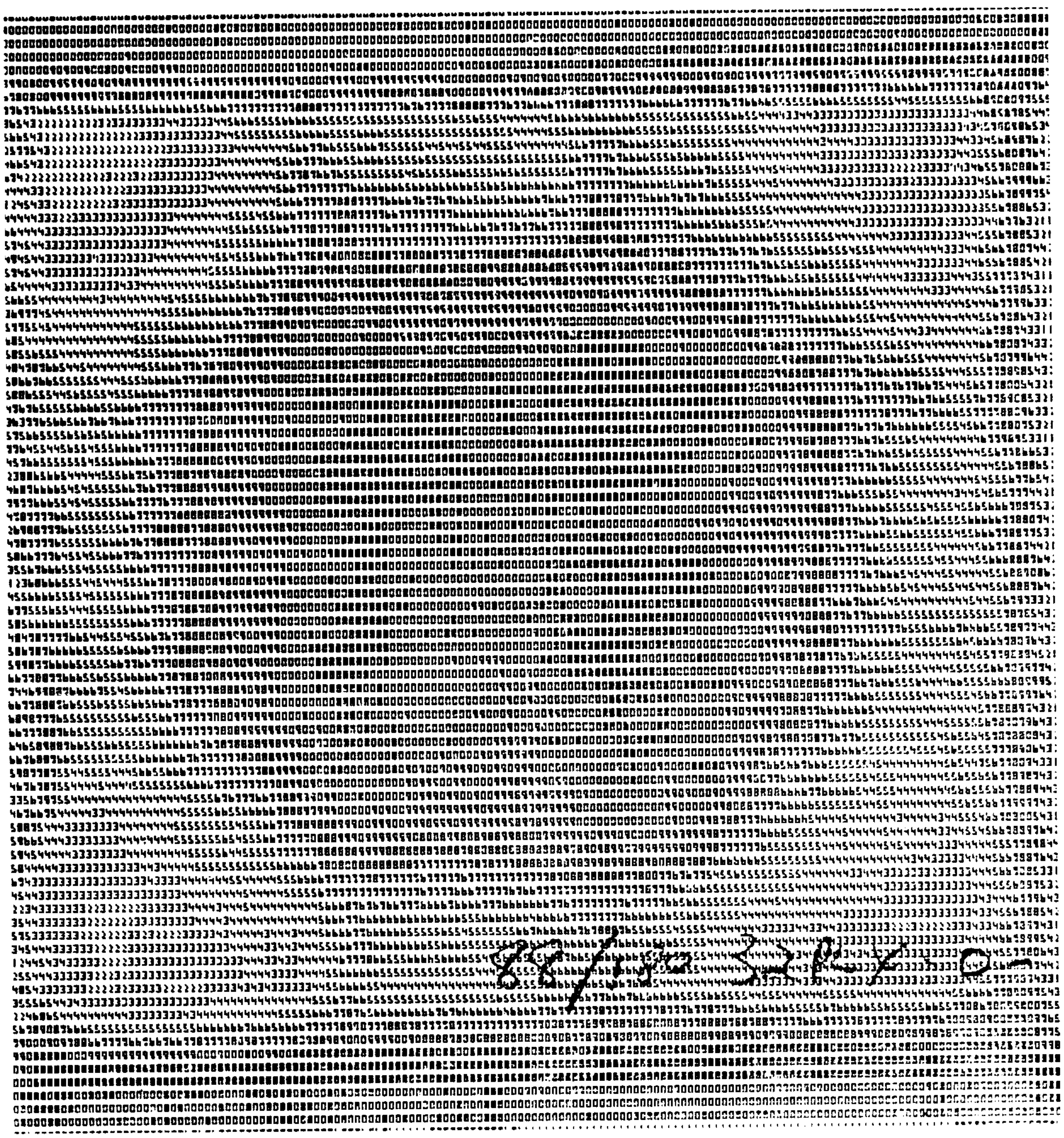


Figure C8. TTU Scan of Laminate 886B5-DT623-02, (0/90)8s

Figure C9. TTU Scan of Laminate 886C1-DT623-02, (0)8

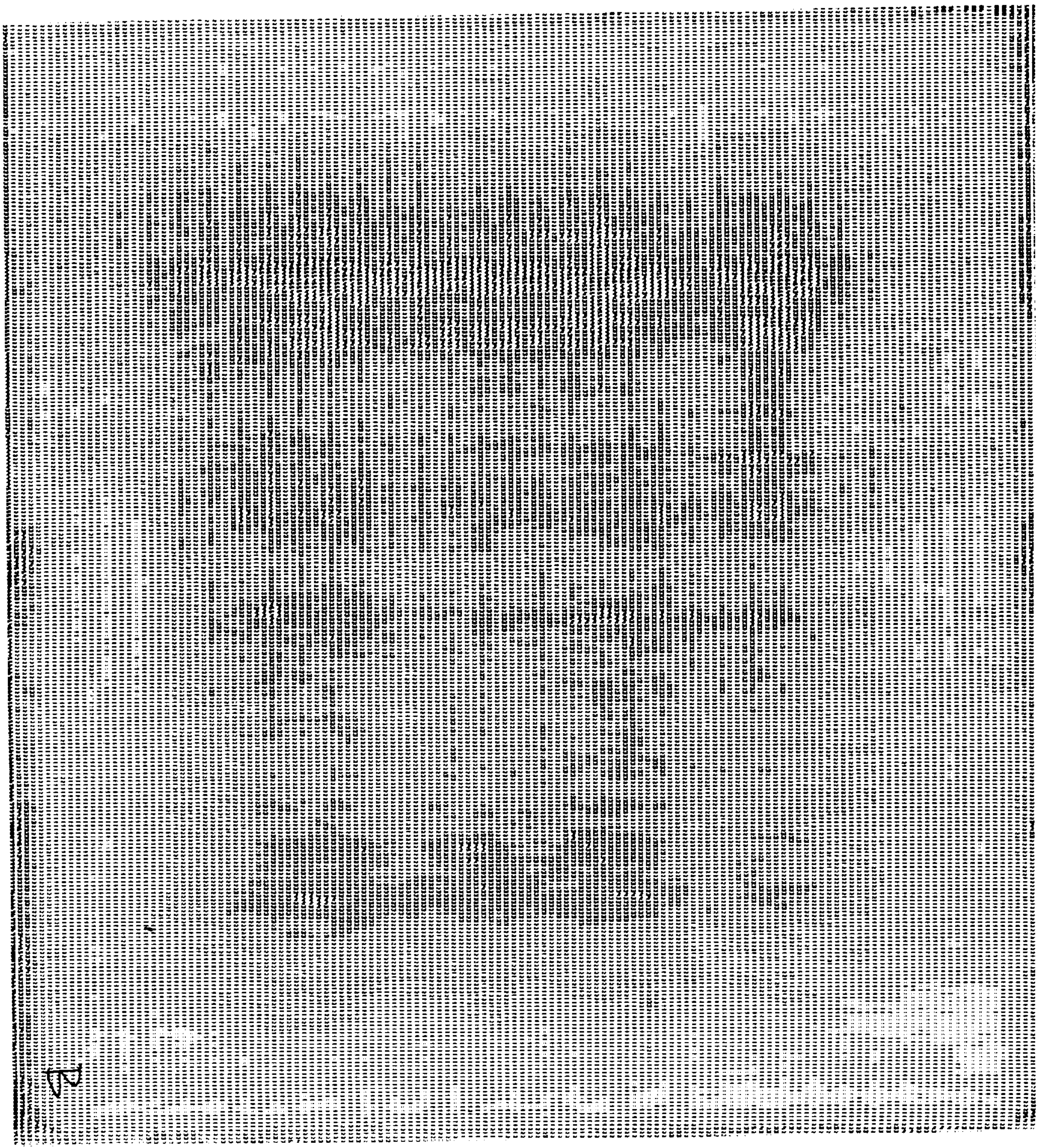


Figure C10. TTU Scan of Laminate 886C2-DT623-02, (0)12

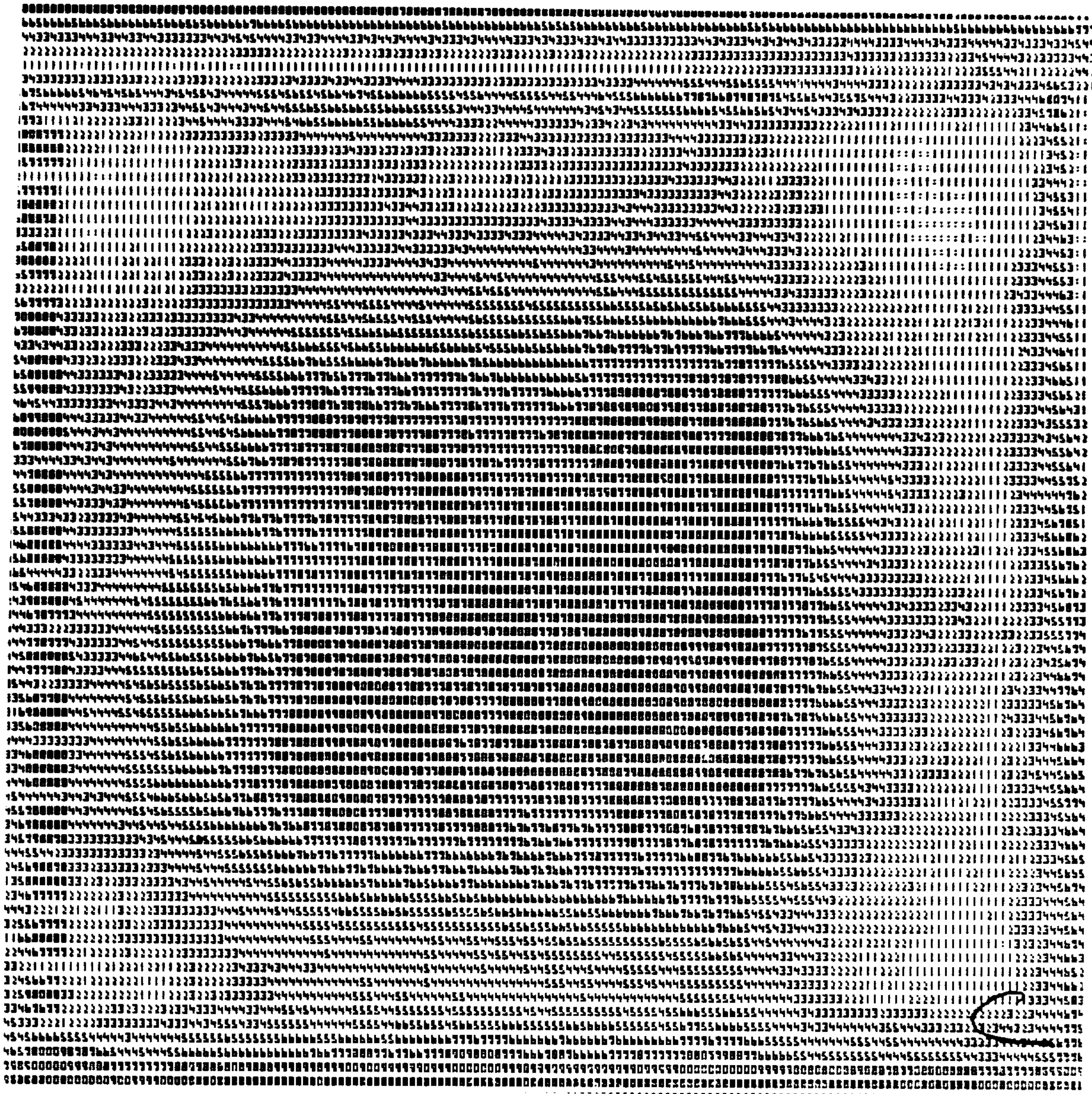


Figure C11. TTU Scan of Laminate 886C3-DT623-02, (0/90)8s

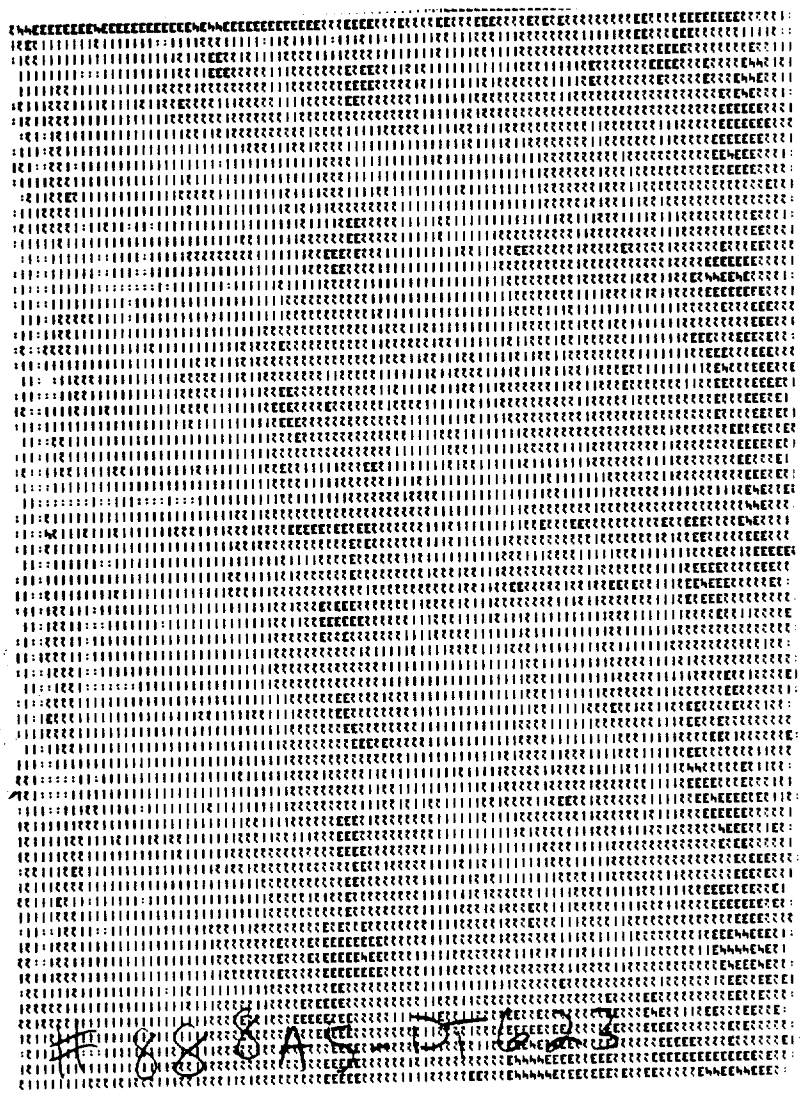


Figure C12. TTU Scan of 32 Ply (0/90) Deg Laminate

Report Documentation Page

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15. Supplementary Notes

Langley Technical Monitor: Paul M. Hergenrother

Final Report - Phase II

16. Abstract

High temperature structural resins are required for use on advanced aerospace vehicles as adhesives and composite matrices. NASA-Langley developed polyimide resins were evaluated as high temperature structural adhesives for metal to metal bonding and as composite matrices. Adhesive tapes were prepared on glass scrim fabric from solutions of polyamide acids of the semicrystalline polyimide LARC-CPI, developed at the NASA-Langley Research Center. Using 6Al-4V titanium adherends, high lap shear bond strengths were obtained at ambient temperature (45.2 MPa, 6550 psi) and acceptable strengths were obtained at elevated temperature (14.0 MPa, 2030 psi) using the Pasa-Jell 107 conversion coating on the titanium and a bonding pressure of 1.38 MPa (200 psi). Average zero degree composite tensile and compressive strengths of 1,290 MPa (187 ksi) and 883 MPa (128 ksi) respectively were obtained at ambient temperature with unsized AS-4 carbon fiber reinforcement.

17. Key Words (Suggested by Author(s)): Thermoplastic, Polyimide, LARC-CPI, LARC-TPI, Adhesives, Composites	18. Distribution Statement: Unclassified-Unlimited Subject Category 27

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